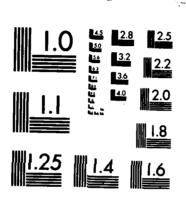
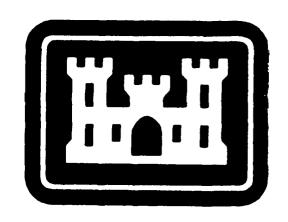
AD-A13 UNCLÄS	POT	OMAC E INEERS	STUAR'	Y E (U) MON NA CA	ITGOMER J M N	RY (JA) IONTGO	EVALU MES M) METRY	CONSL	JLTING 3	E 1/	6
	M											
						c						



MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963-A

U.S. ARMY CORPS OF ENGINEERS BALTIMORE DISTRICT



136865

V

OPERATION, MAINTENANCE AND PERFORMANCE EVALUATION

of the

POTOMAC ESTUARY EXPERIMENTAL WATER TREATMENT PLANT ___.

APPENDIX - VOLUME 1



SEPTEMBER 1980 - SEPTEMBER 1983

This document has been approved for public at a sense and sale; had distribution is unlimited.

JAMES M. MONTGOMERY, CONSULTING ENGINEERS, INC.



SECURITY CLASSIFICATION OF THIS PAGE (When Date Entered)

REPORT DOCUMENTATION F	PAGE	READ INSTRUCTIONS BEFORE COMPLETING FORM
T. REPORT NUMBER MWA-83~WA	AD-A136	BEFORE COMPLETING FORM
PERFORMANCE EVALUATION, MAINTENAMENT PERFORMANCE EVALUATION OF THE POWATER TREATMENT PLANT, EXECUTED Volume, Appendix Volumes 1 and	ANCE AND O'TOMAC ESTUARY - Summary, Main	Final Report 9/80 to 9/83
7. AUTHOR(*) James M. Montgomery, Consulting Pasadena, California	Engineers, Inc	DACW 31-80-C-0041
 PERFORMING ORGANIZATION NAME AND ADDRESS Baltimore District U.S. Army Corps of Engineers, After P.O. Box 1715, Baltimore, Maryla 		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
Baltimore District U.S. Army Corps of Engineer, ATT P.O. Box 1715, Baltimore, Maryla MONITORING AGENCY NAME & ADDRESS(II dillerent)	and 21203	12 REPORT DATE September 1983 13. NUMBER OF PAGES 2329 15. SECURITY CLASS. (of this report) Unclassified 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release, distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)



18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Water Resources; Water Supply; Water Resources Development Act 1974, PL 93-251; Potomac Estuary Experimental Water Treatment Plant; Potomac River; Water Treatment; Contaminated Water Supply; Advanced Water Treatment; Granulated Activated Carbon Adsorption; Ozone Disinfection; Water Treatment Demonstration Plant; Water Reuse; U.S. Army Corps of Engineers; Water Quality Analysis and EPA Dynamic The Water Resources Act

of 1974 authorized the Baltimore District of the U.S. Army Corps of Engineers to investigate the use of the Potomac River Estuary as a possible supplemental water supply source for the Metropolitan Washington Area (MWA). Use of the Estuary, a source expected to be contaminated with substantial amounts of treated wastewater during a severe drought, was one of several structural and non-structural alternatives to meet the long term water supply needs of the MWA, which were evaluated in the U.S. Army Corps of Engineers' MWA Water

DD 1 JAN 73 1473 EDITION OF 1 NOV 65 IS OBSOLETE

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE(When Date Entered)

19. KEY WORDS (continued)

Estuary Model.

20. ABSTRACT (continued)

Supply Study.

The investigation evaluated the water quality produced by a 1.0 MGD demonstration water treatment plant (EEWTP), which was located adjacent to the Estuary at the Blue Plains WPCP, Washington, D. C.

Based on certain hydrologic conditions and the results of the EPA Dynamic Estuary Model, a raw water mix of 50 percent estuary water and 50 percent nitrified Blue Plains sewage effluent was selected for treatment.

Three water treatment process combinations were investigated. The first process combination included alum coagulation, sedimentation, intermediate chlorination, gravity filtration, granular activated carbon (GAC) adsorption and free chlorine disinfection. The second procees substituted ozone as the intermediate oxidant. The final combination consisted of lime coagulation, sedimentation, recarbonation, gravity filtration, GAC adsorption at twice the contact time, ozone and chloramine for final disinfection.

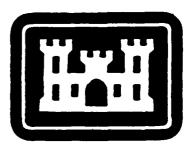
An extensive water quality analysis program was conducted to determine the acceptability of the water for human consumption. The sampling frequency rates exceeded recommended standards. The analytical program parameters included physical and aesthetical (13); major cations, anions and nutrients (19); trace metals (24); radiological (5); microbiological (6) including enteric viruses (41 identifiable types), parasites (7), and four bacterial groups; organic (151); and toxicological (2). Finished water samples were collected from three MWA water treatment plants to compare their water quality against the project's finished water quality.

Within the limits of the analytical techniques used and the influent water quality conditions observed it was concluded that the three process combinations monitored were technically feasible of producing a water acceptable for human consumption.

Estimated treatment cost for a 200 MGD estuary water treatment plant, using design and operating criteria similiar to that used in the EEWTP, are approximately 34.3¢/1000 gallons for the first alum mode and 47.6¢/1000 gallons for the lime mode of operations. Due to uncertainties over the plant's location, intake and certain finished water structures and related costs were excluded from the cost estimates.



U.S. ARMY CORPS OF ENGINEERS BALTIMORE DISTRICT



OPERATION, MAINTENANCE AND PERFORMANCE EVALUATION

of the

POTOMAC ESTUARY EXPERIMENTAL WATER TREATMENT PLANT

APPENDIX - VOLUME 1

SEPTEMBER 1980 - SEPTEMBER 1983



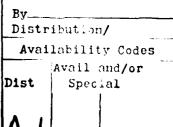
JAMES M. MONTGOMERY, CONSULTING ENGINEERS, INC.



TABLE OF CONTENTS

APPENDICES - VOLUME I	Page
APPENDIX A - SPECIALIZED ANALYSES AND QUALITY ASSURANCE	E A-0-1
Section 1 - Specialized Analyses Protocols	A-1-1
Particle Size Distribution Analysis	A-1-1
Definition	A-1-1
Instrumentation	A-1-1
Asbestos Procedure	A-1-4
Summary of Method	A-1-4
Sampling Methods	A-1-4
Procedure	A-1-4
Counting and Identifying Asbestos Fibers	A-1-5
Bromide by Ion Chromatography	A-1-6
Introduction	A-1-6
General Discussion	A-1-6
Apparatus	A-1-7
Reagents	A-1-7
Procedure	A-1-8
Calculations	A-1-10
Quantitation of Salmonella Sp. in Water	A-1-12
Enrichment	A-1-12
Isolation	A-1-12
Primary Biochemical Screening	A-1-12
Quantitative Determination of Viruses	A-1-13
Virus Adsorption	A-1-13
Elution	A-1-13
Reconcentration	A-1-13
Virus Assay	A-1-14
Virus Identification: Neutralization by	
Cytopathic Effects	A-1-15
Procedure for Using the Dried Lim Benyesh-Melnick Pools	
for Typing Enteroviruses	A-1-16
Parasite Assay	A-1-27
Parasite Sample Collection	A-1-27
Sample Preservation	A-1-27
Sample Processing	A-1-27
Parasite Concentration	A-1-27
Parasite Staining	A-1-28
Parasite Identification	A-1-28 'n For
Ames Salmonella/Mammalian-Microsome Test Method for	A&I
Detecting Mutagens in Water and Wastewater Concentrates	A-1-30
Principles of the Ames Test	A-1-30 ed 🔲
Salmonella Strains	A-1-30 ation
-I	





APPENDICES - VOLUME I	Page
Quality Control Check for the Mutation Integrity of	
Tester Strains	A-1-31
Preparation of Media, S-9 Reaction Mixture	
and Tester Strains	A-1-32
Plate Incorporation Assay	A-1-34
Testing for In Vitro Transformation in the C3H/10T1/2	
Mouse Embryo Fibroblast System	A-1-36
Background	A-1-36
Maintenance and Handling of C3H Mouse Embryo Cell Line	A-1-37
Quality Assurance	A-1-39
Procedures for the Assay	A-1-39
Analysis of Organic Carbon by Photochemical Oxidation/	
Infrared Absorption	A-1-40
Definition	A-1-40
Apparatus	A-1-40
Reagents	A-1-40
Preparation of Standards and Reagents	A-1-40
Analysis	A-1-41
Calculation	A-1-42
Quality Assurance	A-1-42
Analysis of Organic Halogen in Potable and Non-Potable	
Water by Carbon Adsorption	A-1-44
Definition	A-1-44
Apparatus	A-1-44
Reagents	A-1-44
Preparation of Standards and Reagents	A-1-44
Sample Concentration and Analysis	A-1-45
Calculation	A-1-47
Quality Assurance	A-1-47
Analysis of Trihalomethanes in Potable and Non-Potable	
Water by Liquid-Liquid Extraction	A-1-50
Definition	A-1-50
Apparatus	A-1-50
Reagents	A-1-50
Standard Preparation	A-1-50
Internal Standard	A-1-51
Extraction and Analysis	A-1-51
Chromatographic Analysis	A-1-53
Calculation	A-1-53
Quality Assurance	A-1-53
Analysis of Purgeable Organic Compounds in Potable and	
Non-Potable Water by Gas Chromatography Mass Spectrometry	A-1-55
Definition	A-1-55
Apparatus	A-1-55
Reagents	A-1-55
Standard Preparation	A-1-55
Calculation	A-1-57
Quality Assurance	A-1-57

APPENDICES - VOLUME I	Page
Analysis of Base/Neutrals and Acids in Potable and Non-Potable	
Water by GC/MS	A-1-59
Definition	A-1-59
Apparatus	A-1-59
Reagents	A-1-59
Standard Preparation	A-1-59
Internal Standard	A-1-59
Extraction	A-1-59
Analysis	A-1-61
GC/MS System	A-1-61
Calculation	A-1-62
Quality Assurance	A-1-62
Analysis of Herbicides in Potable and Non-Potable Water by GC	A-1-64
Definition	A-1-64
Apparatus	A-1-64
Reagents	A-1-64
Standard Preparation	A-1-64
Internal Standard	A-1-64
Extraction and Esterification	A-1-64
Analysis	A-1-66
Calculation	a-1-67
Quality Assurance	A-1-67
Analysis of Pesticides and PCBs in Potable and Non-Potable	
Water by GC	A-1-69
Definition	A-1-69
Apparatus	A-1-69
Reagents	A-1-69
Standard Preparation	A-1-69
Internal Standard	A-1-69
Extraction	A-1-69
Analysis	A-1-71
GC System	A-1-71
Calculation	A-1-72
Quality Assurance	A-1-72
Analysis of Semi-Volatile Compounds in Potable and Non-Potable	
Water by Closed-Loop Stripping and Gas Chromatography Mass	
Spectrometry	A-1-73
Definition	A-1-73
Apparatus	A-1-73
Reagents	A-1-73
Standard Preparation	A-1-73
Sample Concentration	A-1-74
Analysis	A-1-74
GC/MS System	A-1-74
Quality Assurance	A-1-75
Analysis of Volatile Polar Organics in Potable and Non-Potable	
Water by Steam Distillation and GC/MS	A-1-77
Definition	A-1-77
Apparatus	A-1-77



APPENDICES - VOLUME I	Page
Reagents	A-1-77
Standard Preparation	A-1-77
Distillation	A-1-77
Analysis	A-1-78
Analysis of Organic Anions in Potable and Non-Potable Water	
By Ion Exchange and GC/MS	A-1-79
Definition	A-1-79
Apparatus	A-1-79
Reagents	A-1-79
Standard Preparation	A-1-79
Sample Preparation	A-1-79
GC/MS System	A-1-81
Calculation	A-1-82
Quality Assurance	A-1-82
Analysis of Organic Cations in Potable and Non-Potable Water	
by Ion Exchange and HPLC	A-1-84
Definition	A-1-84
Apparatus	A-1-84
Reagents	A-1-84
Standard Preparation	A-1-84
Sample Preparation	A-1-85
HPLC System	A-1-85
Calculation	A-1-86
Quality Assurance	A-1-86
Analysis of Polynuclear Aromatic Hydrocarbons in Water by	
High Pressure Liquid Chromatography	A-1-88
Definition	A-1-88
Apparatus	A-1-88
Reagents	A-1-88
Standard Preparation	A-1-88
Extraction	A-1-89
Column Chromatography Clean-up	A-1-90
Analysis	A-1-91
Calculation	A-1-91
Quality Assurance	A-1-91
Analysis of Dihaloacetonitriles in Potable and Non-	
Potable Water by Liquid-Liquid Extraction and Gas	4 1 00
Chromatography	A-1-93
Definition	A-1-93
Apparatus	A-1-93
Reagents	A-1-93
Standard Preparation Extraction	A-1-93
	A-1-94
Chromatographic Analysis Calculation	A-1-96
Calculation	A-1-96

ANOS DEFENSE RESERVE LESSESSES LESSESSES SESSESSES

APPENDICES - VOLUME I	Page
Section 2 - Precision and Accuracy Results	A-2-1
Inorganics	A-2-1
Non-Metals (Physical/Aesthetia Anions, Nutrients and	
Major Cations)	A-2-1
Metals	A-2-28
Trace Organics	A-2-58
Section 3 - Quality Control/Quality Assurance	A-3-1
Montgomery Laboratories - Quality Assurance Manual	A-3-1
Introduction	A-3-1
Quality Assurance Organization	A-3-1
Equipment	A-3-2
Sample Control and Reporting	A-3-3
Chain of Custody	A-3-5
General Procedures	A-3-6
Quality Control - Inorganic Chemistry	A-3-7
Quality Control - Asbestos	A-3-14
Quality Control - Radiochemistry	A-3-15
Quality Control - Microbiology - Pasadena	A-3-16
Quality Control - Organic Chemistry	A-3-23
Quality Control - Microbiology - EEWTP	A-3-30
Attachment 1 - Sample Collection	A-3-42
Attachment 2 - Shewart Control Charts	A-3-48
Section 4 - Comparative Odor Testing	A-4-1
Appendix A - References	A-R-1
APPENDIX B - DATA MANAGEMENT	B-0-1
Section 1 - Data Analysis Techniques	B-1-1
Selection of a Distribution Model	B-1-1
Alternative Models	B-1-1
Evaluation of Models	B-1-4
Tests of Probability Models on Data	B-1-6
Statistical Techniques for Evaluating Data and Estimating	
Sampling Requirements	B-1-9
Parameter Estimation: Data all Quantified	B-1-10
Characterization of Process Effuents and Finished Water	B-1-11
Section 2 - Project Data Management	B-2-1
Hardware	B-2-1
Off-Site	B-2-1
On-Site	B-2-1
Software	B-2-2
Database Concept and Design	B-2-2
Data Entry	B-2-5
Data Cuality Control	D_2_6



APPENDICES - VOLUME I			
Section 3 - Parameter Estimation for Partially Sensored Data: Evaluation of the EM Algorithm for an			
Independent Normal Process	B-3-1		
Introduction and Recommendations	B-3-2		
Iterative EM Algorithm	B-3-2		
Test Results	B-3-5		
Appendix B - References	B-R-1		
APPENDIX C - DYNAMIC ESTUARY MODEL DESCRIPTION	C-0-1		
Introduction	C-0-1		
Types of Models	C-0-1		
Development of the Dynamic Estuary Model	C-0-2		
Applications of the Dynamic Estuary Model in the			
Mid-Atlantic Area	C-0-2		
Potomac River Estuary	C-0-2		
Other Estuaries	C-0-3		
Representation of Potomac River Estuary Geometry	C-0-3		
Characteristics of "Link-Node" Network	C-0-3		
Parameters Describing "Link" Characteristics Parameters Describing "Node" Characteristics	C-0-3 C-0-3		
Hydrologic Component	C-0-4		
Governing Equations and Assumptions	C-0-4		
Numerical Solution Technique	C-0-4		
Initial and Boundary Conditions	C-0-4		
Water Quality Component	C-0-8		
Governing Equations and Assumptions	C-0-8		
Numerical Solution Technique	C-0-8		
Initial and Boundary Conditions	C-0-8		
Appendix C - References	C-R-1		
APPENDIX D - PLANT DESIGN CRITERIA	D-0-1		
Water Treatment Facilities Description	D-0-1 D-0-1		
Chemical Handling System Facilities	D-0-1 D-0-8		
Description	D-0-8		
Solids Handling and Chemical Recovery Facilities	D-0-10		
APPENDIX E - PLANT OPERATIONS DATA	E-0-1		
Section 1 - Average Chemical Use and Daily Average Flow Data for Operational Phases IA, IB and II	E-1-1		



APPENDICES - VOLUME I	Page
Section 2 - Summary Report of Reactivation of EEWTP Spent Granular Activated Carbon by ICI Americas Inc.,	
September, 1981 and April, 1982	E-2-1
Section 3 - Inorganic Analyses for Settled Sludge Samples	E-3-1
Section 4 - Performance Results for the Removal of Volatile	
Organic Chemicals by Surface Aeration	E-4-1

LIST OF TABLES

Table		Page
A.1-1	Parasite Identification Criteria	A-1-29
A.1-2	Eagles Basal Medium	A-1-38
A.1-3	Phosphate Buffered Saline	A-1-38
B.1-1	Goodness of Fit Test Results for Phase IA EEWTP Finished Water	B-1-7
B.2-1	Location Codes	B-2-9
B.2-2	Sampler Codes	B-2-13
B.2-3	Parameter Codes	B-2-14
C.0-1	Freshwater Inflow Boundary Conditions at Chain Bridge	C-0-5
C.0-2	Distribution of Total Wastewater Flow Among MWA Wastewater Treatment Plants	C-0-7
E.1-1	Chemical Use Data - Phases IA and Ib	E-1-2
E.1-2	Chemical Use Data - Phase II	E-1-3
E.1-3	Daily Average Flow Data - Phases IA and IB	E-1-4
E.1-4	Daily Average Flow Data - Phase II	E-1-5
E.3-1	Settled Sludge Major Cations, Anions, and Nutrients	E-3-2
E.3-2	Settled Sludge Trace Metals	E-3-3
E.4-1	Surface Aeration Performance Volatile Organic Chemicals (by LLE) Phase I	E-4-2



green veereern wordsom beardern beereesen stossess vonanne

LIST OF FIGURES

Figure		Follows Page
A.3-1	Sample Processing at Off-Site Laboratory	A-3-3
A.3-2	SIMS: Flow Chart	A-3-4
B.1-1	Chloride in EEWTP Finished Water 95 Percent Confidence Bands of Modified K-S Test	B-1-8
B.1-2	Chloroform (VOA) in EEWTP Finished Water 95 Percent Confidence Bands of Modified K-S Test	B-1-8
B.1-3	Nitrate + Nitrite in EEWTP Finished Water 95 Percent Confidence Bands of Modified K-S Test	B-1-8
B.1-4	Gross Beta in EEWTP Finished Water 95 Percent Confidence Bands of Modified K-S Test	B-1-8
B.1-5	Total Coliforms in EEWTP Finished Water 95 Percent Confidence Bands of Modified K-S Test	B-1-8
B. 1-6	Total Organic Halides in EEWTP Finished Water 95 Percent Confidence Bands of Modified K-S Test	B-1-8
B.1-7	Manganese in EEWTP Finished Water 95 Percent Confidence Bands of Modified K-S Test	B-1-8
B.1-8	Arsenic in EEWTP Finished Water 95 Percent Confidence Bands of Modified K-S Test	B-1-8
B.1-9	Total Organic Carbon in EEWTP Finished Water 95 Percent Confidence Bands of Modified K-S Test	B-1-8
B.2-1	Data Management System Indexed Sequential Access Method Concept	B-2-3
B.2-2	Data Flow Through Transfer Process	B-2-4
C.0-1	Segments Comprising Potomac River Estuary Used In the DEM	C-0-3
C.0-2	Potomac Estuary Model Network Segment 1	C-0-3
C.0-3	Potomac Estuary Model Network Segment 2	C-0-3



List of Figures

Figure		Follows Page
C.0-4	Potomac Estuary Model Network Segment 3	C-0-3
C.0-5	Potomac Estuary Model Network Segment 4	C-0-3
C.0-6	Potomac Estuary Model Network Segment 5	C-0-3

LIST OF ABBREVIATIONS

In order to conserve space and improve readability, the following abbreviations have been used in this report:

A area

ACS automatic composite sampler

BNA base/neutral acid extraction

BOD_g 5-day biochemical oxygen demand

cm centimeter

CLS closed-loop stripping

OC degrees centigrade

D.C. District of Columbia

DEM Dynamic Estuary Model

D.L detection limit

D.T. detention time

ECD electron capture detector

EEWTP Estuary Experimental Water Treatment Plant

EPA Environmental Protection Agency

ERL Environmental Research Laboratory

eV electron volt

FID flame ionization detector

ft feet

g grams

G mixing energy

GAC granular activated carbon

GC gas chromatograph

gpd gallons per day

gpm gallons per minute

HERL Health Effects Research Lab

hp horsepower

HP Hewlett Packard

HPLC high performance liquid chromatography

HSDM Homogenous Surface Diffusion Model

IC Ion Chromatograph

List of Abbreviations

ICAP inductively coupled argon plasma

ID inside diameter

JAWWA Journal of the American Water Works Association

JMM James M. Montgomery, Consulting Engineers, Inc.

JWPCF Journal of the Water Pollution Control Federation

KV kilovolts
M moles/liter

MBAS Methylene-Blue Active Substances

µm micrometers

ug/L microgram/liter

ul microliters µmho micromho

MDC minimal detectable concentration

MDL minimum detection limit

MF membrane filter

MFL million fibers per liter

MGD million gallons per day

mg/L milligram/liter

MINC Modular Instrument Computer

mm millimeter

mM millimole/liter

MPI Malcolm Pirnie, Inc.

MPN most probable number

MS mass spectrometer

mw molecular weight

MWA Metropolitan Washington Area

N normal concentration

NAS/NAE National Academy of Science/National Academy of Engineers

nm nanometer

NRC National Research Council
NTU nephelometric turbidity unit

ODCS Operator Data Collection System

P/A precision/accuracy

PDF probability density function

PM preventive maintenance

List of Abbreviations

ppb parts per billion

ppm parts per million

psi pounds per square inch

Q volumetric flow
QA quality assurance
QC quality control

rpm revolutions per minute

RWQTP Routine Water Quality Testing Program

sec seconds

LECELEGO FELECES

SIMS Sample Information and Management System

SOCs synthetic organic chemicals

SPC standard plate count

TAC technical advisory committee

TDS total dissolved solids

THM tribalomethanes

TTHM total trihalomethanes

TOC total organic carbon

TON total organic nitrogen

TOX total organic halide

TPPAM Testing Program for Process Adjustment and Modification

TSS total suspended solids

UV ultra violet (light)

VAX Virtual Access Extension

VOA volatile organic analyses

wt weight

WQ water quality

yr year

APPENDIX A SPECIALIZED ANALYSES AND QUALITY ASSURANCE

SECTION 1

SPECIALIZED ANALYSES PROTOCOLS

PARTICLE SIZE DISTRIBUTION ANALYSIS

DEFINITION

The HIAC PA-720 Automatic Particle Size Analysis System utilizes light blockage to determine particle size distribution. The instrument consists of a sample introduction apparatus, a 2 to 90 µm sensor and a volumetric cylinder. The sample introduction device is a pressurized chamber which uses air to force the liquid through the sensor at a predetermined rate. Particles in suspension flow past a window of known area. A collimated light beam passes through the sample at right angles to the direction of the flow, then through the window to the photocell detector. Whenever a particle goes through the light beam, a portion of the light is blocked, producing a shadow generating a drop in voltage. The extent of the voltage drop correlates to the particle size. Standardizing of the sensor is done with spherical latex beads of a known size distribution. Each voltage drop corresponding to a particle of equivalent circular cross-sectional area is summed in one of 24 channels, the last one for oversized particles between 90 to 120 µm. The microprocessor computes the size distribution parameters such as average particle count, corrected particle count, standard deviation and calculates the cumulative size distribution of the population.

INSTRUMENTATION

The HIAC PA-720 Automatic Particle Size Analysis System includes the PA-720 module, a teleprinter and an automatic bottle sampler.

ANALYSIS

SE COMPANY DESCRIPTION OF THE PASSESS OF THE PASSESS AND THE PASSESS AND THE PASSESS OF THE SECOND TO

Background and Dilution water

Before sample collection, the background water for dilutions should be analyzed and PA dilution bottles set-up.

- Samples collected from sites before the dual media, filter effluent should be diluted. The dilution factor to be used is 51 or 2 mls of sample per 100 mls of background water.
- 2. Mili-Q treated water is used for the background water sample. The water shall be treated to a quality of 10 Mohm-cm or better.

Whenever Milli-Q is being used, this procedure is to be followed:

- 3. Rinse the PA bottles with Milli-Q water making sure the mouth of the bottle is thoroughly rinsed and is not touched once rinsed.
- 4. Rinse the volumetric flask with Milli-Q water and be sure not to handle the mouth of the flask.
- 5. Fill the flask up to the 100 ml mark and pour into a PA bottle which has been rinsed for dilution. Place a piece of parafilm over the mouth of each bottle. Repeat for all the PA dilution bottles.
- 6. Fill the rinsed PA bottle for background with the Milli-Q and also cover with parafilm
- 7. Run three analyses of the background sample and print the data. Push the ESC button and type STORE to save the data for use as background.

Sample Collection Technique

THE REPORT OF THE PROPERTY OF THE PARTY OF T

- 1. Samples should be collected immediately before they are anlayzed.
- 2. Rinse wide-mouth bottle with sample water making sure the lip is thoroughly rinsed. Do not touch the mouth of the bottle once it has been rinsed.
- 3. Grab a sample with the side-mouth bottle and rinse out the PA bottle thoroughly. Rinse the lip last and do not touch the mouth area afterwards.
- 4. Grab a sample with the wide mouth bottle and pour into the PA bottle. Cover the mouth of the bottle with parafilm and be sure you only touch the sides of the bottle.
- 5. Repeat steps 2 through 4 until all samples are collected.

Dilution Preparation

- 1. The diluted samples should be analyzed first.
- 2. While being analyzed, the diluted samples should be stirred on a setting of 5. Before analyzing, let the sample stir for approximately one minute.
- 3. Analyze three times, if possible, then print out the results.
- 4. Suck air through the sensor between sample analyses to remove residue from the previous sample.
 - a. Close analyzing module.
 - b. Flip AUTOMATIC-MANUAL switch to manual.
 - c. Turn lever on graduated collecting column down.
 - d. Allow to run about 30 seconds.

- 5. Repeat steps 2, 4, 5 and 6 until all diluted samples have been analyzed. When transferring the stirring bead, use the tweezers as much as possible. Remember to rinse both bead and tweezer with Milli-Q between samples.
- 6. The undiluted samples are to be analyzed last.

DESTRUCTION DESCRICTOR (SERVICES DESCRICTOR SERVICE) REPORTS REPORTS DESCRICTOR DESCRIPTION OF SERVICE DESCRIPTION

ASBESTOS PROCEDURE

SUMMARY OF THE METHOD

ALTERNATION SERVICES SERVICES

For the analysis of asbestos in water, the off-site laboratory used the methods described in "Interim Method for Determining Asbestos in Water," (EPA-600/4-80-005). A variable, known volume of water sample was filtered through a 0.1 µm Nuclepore filter to trap asbestos fibers and the filter was then carbon coated. A small portion of the carbon coated filter, with deposited fibers, was placed on an electron microscope grid and the filter material removed by gentle dissolution in chloroform. The material remaining on the grid was examined with a transmission electron microscope (TEM) at a magnification of 20,000. The number of asbestos fibers in twenty grid squares were counted. The asbestos fibers were identified by their morphology and electron diffraction patterns and the length and width of each fiber was measured. The concentration in MFL (million fibers per liter) was calculated from the number of fibers counted, the amount of water filtered, the effective filtration area and the area counted under the TEM. The mass per liter was calculated from the assumed density and the volume of the fibers.

The detection limits were variable and depended upon the variables sited in the preceeding paragraph as well as the amount of total extraneous particulate matter in the sample and the contamination level in the laboratory environment. Under favorable circumstances, 0.1 MFL and total mass of 0.1 ng/L could be detected.

SAMPLING METHODS

The sampling container was a clean, 1 liter, screw-capped polyethylene bottle. Bottles were filled to within two inches of the top to allow for shaking the sample.

No preservatives were added during sampling. The sample was filtered at the off-site laboratory within 48 hours of its arrival.

PROCEDURE

Prior to filtration, the sample was sonicated in an ultrasonic water bath and then shaken. A suitable aliquot ranging from 1 to 100 ml was filtered by vacuum pressure through a 47 mm, 0.1 μ m pore size Nuclepore filter backed by a 0.45 μ m mixed cellulose acetate and cellulose nitrate membrane filter. If the filter contained large amounts of organic material, it was ashed in a low temperature plasma asher to remove the organic material. After ashing, the sample was refiltered to deposit the ashed material onto a 0.1 μ m filter.

After filtration, the filter was removed and placed in a covered petri dish for drying. After the filter had dried, several rectangular strips (15 X 3 mm) were cut from the membrane and fixed on a glass slide for carbon coating. The glass slide was placed in a vacuum evaporator and a 30 to 50 nm layer of carbon was deposited on the filter section. The carbon was evaporated in short bursts to prevent overheating the surface of the filter. Three portions (approximately

2mm²) were cut from the carbon coated filter, placed on separate copper specimen grids supported on copper screening, and placed in a modified Jaffe washer. Chloroform was added to the washer, and the grids were left to sit in the washer for 24 hours. After 24 hours of chloroform treatment, the Nuclepore filter material dissolved, leaving the deposited fibers on the specimen grid. The grids were removed from the washer and placed on a spot plate to evaporate any residual chloroform.

COUNTING AND IDENTIFYING ASBESTOS FIBERS

The grids were examined using a Zeiss EM-10 transmission electron microscope. The microscope was capable of operating between 20 and 100 kV with a magnification up to 200,000, and of carrying out selected area electron diffraction on an area of approximately 0.3 µm².

The grids were examined at low magnification to see that the fibers were uniformly distributed, and that the proper amount of material was deposited on the filter. If the distribution was satisfactory, twenty grid squares from two grids were examined at 20,000 magnification. Identification of asbestos fibers was confirmed by their characteristic morphology and electron diffraction pattern. After counting the fibers present in twenty grid squares, the fiber concentration was calculated using the following formula:

$$C = \frac{(\mathbf{F} \times \mathbf{A_f})}{(\mathbf{Ag} \times \mathbf{V_O})} \times 1000$$

where:

STATES CONTROL CONTROL CONTROL

ANGERTAL WINDSHIPS MAKENAM (MAKENIM MAKENAM

C = Concentration of Fibers (MFL)

F = Average Number of Fibers per Grid Opening

 $A_f = Effective Filtration Area of Filter Paper (mm²)$

Ag = Average Area of One Grid Square (mm²)

V_O = Original Volume of Sample Filtered (ml)

BROMIDE BY ION CHROMATOGRAPHY

INTRODUCTION

In many water systems it may be desirable to determine the concentrations of all or some of the common anions (F, CL, NO₂, PO₄, BR, NO₃, SO₄) in order to assess the need for specific treatment (e.g., fluoridation, nitrate removal, TDS removal) or to generally characterize the water.

To efficiently apply traditional methods of analysis for quantification of individual anions (e.g., colorimetric, electrometric, or titrimetric methods) it is helpful to have prior knowledge of sample constituents. Since Ion Chromatography (IC) is a powerful qualitative and quantitative technique, it is useful in screening a wide variety of water types in which the relative concentrations of anions are unknown. IC eliminates the use of hazardous reagents such as brucine or cadmium used for nitrate testing and readily distinguishes between halides (F, Cl, Br) and oxide forms (SO₃/SO₄ and NO₂/NO₃).

This method is an adaptation of the general IC method proposed for inclusion in Standard Methods for the Examination of Water and Wastewater.

GENERAL DISCUSSION

- 1. Principle: A water sample containing various anions (F, Cl, NO₃, SO₄, etc.) is injected into a stream of carbonate/bicarbonate eluent and passed through a series of ion exchangers. The anions of interest are separated based on their relative affinities for a low capacity strongly basic anion exchanger (guard and separator column). The separated anions are directed onto a strongly acidic cation exchanger (suppressor column) where the anions of interest are converted to their highly conductive acid form (e.g., HF, HCl, HNO₃, H₂SO₄) and the carbonate/bicarbonate eluent is converted to carbonic acid, a low conductive species. The separated anions in their acid form are measured by conductivity. Anions are identified based on their retention times compared to known standards. Quantitation is based on measurement of peak area or peak height.
- 2. Interference: Any substance present in a water sample with a retention time which coincides with that of the ions of interest is a potential interference. Examples are the presence of large amounts of small organic acids interfering with chloride or fluoride determination or the presence of large amounts of one ion interfering with the resolution of another one (for instance NO2 interfering with Br or SO4 interfering with NO₃). Other sources of interference are the effect of one constituent on the response factor of another. Many of these interferences can be overcome by sample dilution. If there are any questions about the identification and quantitation of a peak the sample should be quantitated by the method of known addition. In addition any contaminants present in the reagent water, glassware, or sample processing apparatus (e.g., syringe) can provide spurious peaks. Due to the small volume of sample required for ion chromatography, contamination must be scrupulously avoided.

3. Minimal detectable concentration: The minimal detectable concentration (MDC) for a given anion is a function of the sample size chosen and the conductivity scale used. In general MDC's are in the range of 0.05 mg/L for F, and 0.1 mg/L for Cl, NO₂, O-PO₄, Br, NO₃, and SO₄ using a 100µ sample loop and a 3µmho fullscale setting on the conductivity detector. Similar values may be achieved by using a higher scale setting and an electronic integrator.

To optimize the method for Br, a 300 µl sample loop and a 0.3 µmho fullscale setting on the conductivity detector were used.

APPARATUS

- 1. Ion chromatograph capable of delivering 2 to 5 ml of eluent per minute at a pressure of 200 to 1000 psi. (Note 1) The IC shall be equipped with an injection valve, a sample loop; (Note 2) guard column, and separator and suppressor columns. In addition it shall be equipped with a temperature compensated small volume conductivity cell (6 µl volume or less), and a strip chart recorder capable of full scale response of 2 sec or less. An electronic peak integrator is optional.
- 2. Anion separator columns-low capacity pellicular anion exchange resin that is styrene divinylbenzene-based and is capable of resolving F, Cl, NO₂, PO₄, Br, NO₃, and SO₄. 4x 250 mm, DIONEX P/N 030827 (normal run) or DIONEX P/N 030831 (fast run) or equivalent.
- 3. Anion guard column-same as separator but shorter column to protect the separator column from being fouled by particulates or certain organic constituents. 4 x 50 mm, DIONEX P/N 030825 (normal run) or DIONEX P/N 030830 (fast run) or equivalent.
- 4. Anion suppressor column-high capacity cation exchange resin capable of converting eluent and separated anions to their acid forms. DIONEX P/N 030828 (requires regeneration) or DIONEX P/N 35350 (fiber suppressor-continuously regenerated) or equivalent.

REAGENTS

- Reagent water: deionized or distilled water in which an interferent is not observed at the method detection limit of each parameter of interest.
- 2. Eluent solution: Standard, 0.003 M NaHCO₃, 0.0024 M Na₂CO₃. Dissolve 1.008 g NaHCO₃ and 1.0176g Na₂CO₃ in reagent water and dilute to 4 liters.
- 3. Regenerant Solution 1. 1N H₂SO₄: Cautiously add 111 mls concentrated H₂SO₄ (36 N) to approximately 600 ml of reagent water. Cool and dilute to 4 L with reagent water. This solution is required when the suppressor used is not a continuously regenerated type.

- 4. Regenerant Solution 2. 0.025N H₂SO₄: Dilute 2.8 ml concentrated H₂SO₄ to 4 L with reagent water. Alternatively dilute 100 mls Regenerant Solution 1 to 4 L with reagent water. This solution is required for continuous regeneration of fiber suppressor when that system is used.
- 5. Stock standard solutions-1000 mg/L. Dry all standard material except NaNO2 to a constant weight at 105°C and cool before weighing.

Parameter	Standard Material	
Chloride (Cl)	NaCl	1.6485 g/L
Fluoride (F)	NaF	2.2100 g/L
Bromide (Br)	NaBr	1.2876 g/L
Nitrate (NO ₃)	NaNO ₃	1.3707 g/L
Nitrite (NO2)	NaNO ₂	1.4998 g/L (dessicate to constant wt)
Phosphate (PO ₄)	KH ₂ PO ₄	1.4330 g/L
Sulfate (SO ₄)	K2SO4	1.8141 g/L

To prepare these individual standards dilute each of the weighed standard materials to 1L using reagent water. Store in plastic bottles.

6. Combined working Standard Solution 1. (high range). 10 mg/L F, 10 mg/L Cl, 10 mg/L NO₂, 1 mg/L Br, 10 mg/L NO₃, 10 mg/L PO₄, 100 mg/L SO₄.

Prepare by diluting 10.00 ml each of the 1000 mg/L stock standards for Cl, F, NO₂, NO₃, and PO₄, 1.00 ml of the 1000 mg/L stock Br standard and 100 ml of the 1000 mg/L stock SO₄ standard to 1000 ml with reagent water. Store in a plastic bottle protected from light.

7. Combined working Standard Solution 2. (low range) 1 mg/L F, 1mg/L Cl, 1 mg/L NO2, 0.1 mg/L Br, 1 mg/L NO3, 1 mg/L PO4, 10 mg/L SO4.

Prepare by diluting 100 ml of combined working Standard Solution 1 to 1000 ml with reagent water. Store in a plastic bottle protected from light.

8. Alternate combined working standard solutions.

Prepare by diluting appropriate quantities of the stock standards with reagent water. These standards should be representative of the concentration levels anticipated in the samples. They may be either higher or lower in range than those suggested here.

Note: Concentrated stock solutions are stable for at least one month if kept refrigerated. Working standard solutions should be prepared fresh daily if NO_2 or PO_4 are to be determined. If these components are not part of the combined standards, working standards are stable for a month.

PROCEDURE

1. System equilibration: The IC is turned on and the eluent flow rate adjusted to produce the desired peak separation. For older instrumenta-

tion (DIONEX models 10-16) the required flow rate is approximately 2.3 mls/min (pressure 400-500 psi). The detector is adjusted to the desired setting (typically 10 mho) and the system allowed to equilibrate (15-20 minutes usually suffices). The system is equilibrated when the baseline stabilizes. The detector offset is then adjusted to zero out the conductivity of the eluent. (Note 3)

2. Determination of retention times and calibration: Approximate retention times can be determined and recorded by injecting standards containing only the element of interest or by injecting the combined standard and determining the individual retention times. Actual retention times will vary as a function of the separator column used, concentration of the individual ion, temperature, and eluent flow rate. However retention will always be in the order F, Cl, NO2, PO4, Br, NO3, SO4 when the standard eluent and anion separator column is used.

Initial calibration is performed by injecting a minimum of three (3) different concentration standards made from various dilutions of the working standards. A calibration curve is constructed by plotting peak height or peak area versus concentration on linear graph paper. Any time the detector setting is changed standards must be injected at that setting unless it has been previously verified that linearity extends through several settings (Note 4, 5).

Once linearity has been demonstrated for a given detector setting, calibration can be performed using only one standard injection for a given detector setting. The peak height (or area) and retention time for a given concentration standard is recorded to allow calculation of a calibration factor (F).

3. Injection of samples: Prior to injection any sample containing particulates >0.2 µm must be filtered to avoid deposition of particulates which eventually restrict eluent flow. A sample or standard is injected into the ion chromatograph using a prewashed small syringe (1-10 mls) equipped with a male luer fitting. Enough sample is injected to flush the sample loop several times (for a 0.3 ml sample loop at least one (1) ml is injected).

The IC is then switched from the load to inject mode and the peak heights and retention times recorded on a strip chart recorder. When the last peak (SO₄) has appeared and the conductivity signal has returned to baseline, a new sample can be injected by repeating the process.

If the measured peak height or peak area exceeds the calibrated range of the system, dilute the sample with an appropriate amount of reagent water (or eluent) and reanalyze.

4. Regeneration (for systems not equipped with fiber suppressor): When the suppressor column becomes partially exhausted (indicated by conductivity increase to stable value of over 300 µmho), regenerate with 1N H₂SO₄ in accordance with manufacturer's instructions.

CALCULATIONS

Prepare calibration curves for each anion of interest by plotting peak area or peak height units of standards against concentration values. Compute sample concentration by comparing sample peak height or area with the standard curve. Alternatively, when response is shown to be linear it is possible to compute concentrations using the equation:

 $C = H \times F \times D$

where

C = concentration in mg/L for an individual anion

H = peak height or area

F = response factor, calculated as concentration of

standard/height (or area) of standard

D = dilution factor for samples requiring dilution

PRECISION AND ACCURACY

In a single laboratory 6 natural water samples containing low levels of Br (.027 to 1 mg/L) were analyzed by this method. Average precision (7 replicates each) was 3.2 percent and the average recovery for spikes was 96 percent. The calculated detection limit (3 sigma error) was .003 mg/L.

NOTES

- 1. The Dionex Model was used in this project.
- 2. Loop size can be varied with varying sensitivity. Precision and accuracy data shown here are based on a 300µl loop. Greater sensitivity can be acheived with larger loops and a wider dynamic range acheived with smaller loops. The only precaution to observe in this respect is not to exceed the capacity of the separator column.
- 3. With the fiber suppressor the regeneration flow rate should be adjusted to maintain a stable baseline (typically 2.5 to 3 ml/min).
- 4. With a system requiring suppressor regeneration, NO₂ interaction with the suppressor can produce slight errors in NO₂ determination. This determination should only be performed when the suppressor is at the same stage of exhaustion as when the standard is injected. Otherwise NO₂ signals will gradually increase through the course of a run. In that case the system should be restandardized frequently throughout a days run. Such an interaction is not observed with systems using a fiber suppressor.
- 5. With a system requiring suppressor regeneration the water dip may shift slightly during exhaustion of the suppressor. When a fast run column is being used this may result in a slight interference with the Cl or F peaks at low concentrations. If the water dip interferes, the estimated sample concentration must be bracketed by analyzing standards at a similar concentration. To compensate for this potential interference it is possible to eliminate the water dip by either a) diluting the sample with eluent if sample dilution is required before analysis or b) adding an equivalent of 1.0 ml of a prepared eluent concentrate (solution that is 100 times more concentrated than the eluent used for analysis) per 100 ml of sample. When the sample is prepared in this manner it is important to

prepare standard solutions and blanks the same way to compensate for any ionic impurities present in the eluent.

ATOTAL PERSONAL STANDERS - FEBRUAR - INCOPPERSON - AND STANDERS - STANDERS - STANDERS - STANDERS - STANDERS - AND STANDERS - AND STANDERS - AND STANDERS - STANDERS - STANDERS - AND STANDERS - AND STANDERS - AND STANDERS - STANDERS - AND STANDERS

QUANTITATION OF SALMONELLA SP. IN WATER

ENRICHMENT

Dulcitol-selenite broth was prepared and distributed into sterile bottles on the day of analysis. Each bottle contained 50 ml of medium and either 10 ml raw water or 1,000 ml of finished water (filtered through a 0.45 µm Gelman GN-6 filter). After filtration the filter was aseptically removed and transferred to a bottle containing enrichment medium. Ten ml samples were directly inoculated into the enrichment medium. Five replicates were filtered for each sample. The bottles were incubated for three days at 35°C. At 24-hour intervals, they were examined for turibdity and an orange-red color due to the reduction of selenite. The positive broth cultures were streaked onto selective solid media for confirmation and isolation.

ISOLATION

The solid medium used for isolating <u>Salmonella</u> sp. from the enrichment broth cultures was xylose lysine desoxycholate (XLD) agar. <u>Salmonella</u> and <u>Arizona</u> organisms produce black-centered red colonies on this medium, whereas many coliform bacteria, such as <u>Enterobacter</u> and <u>Proteus</u> produce yellow colonies. Colonies typical of <u>Salmonella</u> sp. were purified by restreaking onto XLD plates.

PRIMARY BIOCHEMICAL SCREENING

Three types of primary identification media were utilized for <u>Salmonella</u> isolates. These were triple sugar iron (TSI) agar, urea agar (Christensen) and lysine iron agar (LIA).

TSI slants were inoculated by stabbing the butt and streaking the slant. The tubes were incubated at 35°C for 10 to 24 hours. Typical reactions for Salmonella are:

Slant Alkaline (red)

Butt Acid (yellow), gas, H₂S

LIA slants were inoculated by stabbing the butt twice and streaking the slant. The tubes were incubated for 24 to 48 hours at 35°C. Typical reactions for Salmonella are:

Slant alkaline (blue-purple)

Butt alkaline (blue-purple), H₂S + (-)

Urea agar was inoculated by heavily streaking the slant. The tubes were incubated for 18 to 24 hours at 35°C. Cultures of Salmonella are urease negative (yellow) whereas Proteus are urease positive (red).

A five tube MPN test was used to quantitate <u>Salmonella</u>. A positive tube was one from which an isolate was identified as <u>Salmonella</u> after primary biochemical screening.



VIRUS ADSORPTION

The virus concentration method was based on an adsorption/elution procedure described in the 14th edition of Standard Methods. Conditions were adjusted so that viruses first adsorbed and then were eluted from a filter. This required that the sample be adjusted to pH 3.5 and Al (III) added to obtain a final concentration 0.005 M (influents) or 0.0005 M (effluents). These chemical additions are presumed to alter the viral charge to a positive one, causing it to adsorb onto a negatively-charged filter surface (0.45 µm pore size).

ELUTION

The sorbed viruses were eluted twice with one liter of one percent beef extract/0.4 percent glycine at pH 9.0. The filter was initially eluted in a co-current manner to elute the bulk of the non-solids-associated viruses. After collecting this eluate and readjusting the pH to 9.0 with sodium hydroxide, the filter was re-eluted in a counter-current manner. This recovered the filtered solids-associated virus. The concentrate was then transported off-site for reconcentration.

RECONCENTRATION

The eluate was reduced in volume by an organic flocculation technique developed by Katzenelson et al., (1976). The eluate was adjusted to pH 3.5, which approaches the isoelectric point of many enteroviruses and proteins, causing the proteins and viruses to coagulate and flocculate. After allowing the coagulation and flocculation to occur for thirty minutes, the eluate was centrifuged to pellet the proteins, solids, and viruses. The pellet was then resuspended in approximately 30 ml of 0.15 M sodium phosphate buffer, pH 9.0. The resulting solution was treated with antibiotics, incubated at 35°C for one and one half hour and frozen at -20°C. The incubation was necessary to allow the bacteria to grow and become susceptible to antibiotics. Bacteria and fungi growing in the presence of antibiotics lose their cell wall integrity, thereby allowing subsequent freezing at -20°C to kill the microorganisms. This reduced the microbial interference during the virus assay procedure.

To prevent non-specific toxicity, each concentrate was thawed and treated with an equal volume of chloroform. The mixture was shaken for fifteen minutes and then centrifuged for fifteen minutes at 2000 rpm to separate the layers. The supernatant was aspirated and aerated for 30 minutes with filtered N₂ gas to remove any residual chloroform. The concentrate was frozen at -70°C until assayed.

VIRUS ASSAY

The more sensitive procedure for virus isolation is viral infection of a cell monolayer maintained under liquid medium. In this method a gradual and progressive destruction of cells occurs due to the virus infection and is observed and scored microscopically by the virologist. This is generally referred to as the cytopathic effect method (CPE) which was used in this study.

Though this procedure has the advantage of simplicity, it seldom demonstrates the presence of more than one type of virus in a given inoculum. These phenomena can be explained by viral interactions within the liquid medium. Infection and destruction of the cell population can significantly reduce available cells for infection by another virus or the replication process of one virus may be inhibited by another. If the inoculum contains few infective viruses, interference problems are of little consequence, which is the usual case when examining environmental samples.

An additional limitation is that the method normally is restricted to qualitative determinations. A semi-quantitative method, a most probable number (MPN) type sample titration, has been used to estimate virus concentrations (Chang, et al., 1958). Despite these drawbacks, this method is reported to be more sensitive than the plaque assay isolation procedure because many viruses which produce CPE in liquid culture do not consistently plaque out.

The concentrates used for virus isolations were inoculated into liquid culture flasks. Approximately one-half of the concentrate (2 to 5 ml) was assayed on BGM and MA 104 cells. Many enteric viruses are capable of infecting at least one of these cell lines. RD cells were used only during the first few months of monitoring.

The bottles were incubated for ninety minutes at 35°C to allow for the viruses to adsorb to the cells. An evenly dispersed inoculum was assured by rocking the bottles every twenty minutes. The inoculum was poured off and the cell sheet was rinsed with phosphate buffered saline. Maintenance medium was then added to each flask. The bottles were periodically scored for CPE from two to fourteen days postinoculation.

At the end of the incubation period, all bottles were blind passed. In this assay system a blind passage was required as a quality assurance step to screen out the possibilities of missing slow growing viruses and scoring non-viral CPE. A blind pass consisted of transferring 0.5 ml of the liquid from each initial isolation culture to a new tube containing cells of the same type to confirm the initial isolation. The samples were filtered through a 0.22 µm filter to eliminate bacterial and fungal contamination. If the CPE was caused by toxicity the blind passage diluted the sample so that it no longer elicited a toxic response. If there was a slow virus or very low number of viruses, the incubation period on new cells permitted their detection and isolation. These tubes were incubated at 35°C on a roller apparatus and scored from two to fourteen days postinoculation for CPE.

A second blind passage was made after the incubation period. A positive isolation was one which produced typical viral CPE in the second passage.

The sample results were calculated according to the formula below using the CPE negative tubes from the second passage:

$$D = -1/V \ln (S/N)$$

where

D = Most Probable Number Cytopathic Effect Units (MPNCU)/ml concentrate

V = ml of sample concentrate inoculated per tube

S = the number of negative tubes

N = the number of tubes inoculated

The values obtained for D and the volume concentrated were used to back calculate the MPNCU/gallon.

VIRUS IDENTIFICATION: NEUTRALIZATION BY CYTOPATHIC EFFECTS

Positive tubes were frozen at -70°C until the isolates were identified. The Lim Benyesh-Melnick cross-secting antisera were used to identify the virus isolates. Both the plaque and liquid culture methods were used for the neutralization tests. See the following document for a complete description of the analytical procedure involved.

April 1972



RESEARCH RESOURCES BRANCH NATIONAL INSTITUTE OF ALLERGY AND INFECTIOUS DISEASES DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE

PUBLIC HEALTH SERVICE NATIONAL INSTITUTES OF HEALTH BETHESDA, MARYLAND 20014

PROCEDURE FOR USING THE DRIED LIM BENYESH-MELNICK POOLS FOR TYPING ENTEROVIRUSES

The Lim Benyesh-Melnick pools (A-H) consist of 42 equine antisera (1-3) combined into 8 pools, each pool containing 10-11 antisera (4-6). The pools have been designed in such a manner that a given antiserum appears either in one pool, or in two pools or in three pools. Thus, an unknown enterovirus may be identified by its neutralization by the pool or pools containing its homotypic antiserum.

The eight pools prepared in 1971 under NIH sponsorship correctly identify 37 enteroviruses; polioviruses 1-3; coxsackievirus A types 7, 9 and 16; coxsackievirus B types 1, 2 and 4-6; and echoviruses 1-7, 9, 13-21, 24-27 and 29-33.

The identification table is attached.

Due to the inadvertent use in 1971 of certain antisera that had been improperly labeled, identifications of coxsackievirus B-3 and echoviruses l1 and 12 require Special Tests. These are described under Interpretation (paragraph 3a) and may be run simultaneously with the A-H pool tests, or preferably later if so indicated from results of the A-H pool tests. Echoviruses 22 and 23 need be identified only as E22/E23 complex, for they form a closely related subgroup. See Interpretation, paragraph 3b.

1. Rehydration of serum pools. If dried serum pools (A-H) are supplied, each vial should be rehydrated with 5 ml of sterile distilled water to make the undiluted stock. For use in the test, each pool is further diluted 1:10, employing Melnick's medium B containing antibiotics as the diluent. After dilution, 0.1 ml of the pool contains 50 antibody units of each antiserum

2. The procedure for performing the test is as follows:

Step 1. The 1:10 diluted pools (A-H) are distributed into test tubes, 0.3 ml per tube, and the sera inactivated by heating at 56°C for 30 minutes.

Step 2. To each tube is added 0.3 ml of a dilution of the unknown virus containing from $10^{3.0} \pm 0.5$ TCD_{50} 's per 0.1 ml. (Melnick's medium B is the preferred diluent). The serum-virus mixtures are then incubated at 37°C for 2 hours. A sample of the virus dilution used is similarly incubated and subsequently titrated to determine the virus dose employed in the test.

Step 3. Following incubation, two tissue culture tubes are inoculated with each serum-virus mixture, 0.2 ml per tube. The challenge virus dilution is inoculated into each of 4 tissue culture tubes, 0.1 ml per tube, and subsequently titrated using 4 tubes per dilution.

The inoculated tubes are incubated at 37°C for a period of 7 days.

Step 4. Microscopic readings for cytopathic effect (CPE) are made on the tissue culture tubes beginning on the second day of incubation.

As soon as the virus control tubes show CPE to a degree of 3 to 4+ (i.e., CPE involving 75% or more of the cultures), daily readings on the entire test should be made.

Interpretation. The results of neutralization are checked against the scheme for identity (see identification table). Often, a tentative identification can be made on the 4th or 5th day, particularly in instances where the dose of virus is high or the virus grows rapidly. Slower-growing viruses may require the full 7 days of incubation. Complete neutralization on the 7th day with a dose of virus between $10^{2.5}$ and $10^{3.5}$ constitutes a firm identity (see Protocol 1).

There are some instances, however, in which there is complete neutralization on the 4th or 5th days and evidence of a slow breakthrough on the 6th or 7th days, probably due to small aggregates of the virus in the virus culture (7) (see Protocol 2). This may occur especially with field isolates of echoviruses 9, 17 and 27 tested by the pools made in 1971. In this situation, identification is considered adequate, although in rare instances the investigator may deem it advisable to confirm the identification by the use of type-specific antiserum.

Examples of both types of results are shown in the attached protocols.

- a. Special Tests (2 ml vials of dried sera are provided for identification of coxsackievirus B-3 and echoviruses 11 and 12). The 50 unit materials should be stored frozen between uses as well as any unused portion of the rehydrated serum.
 - (i) Coxsackievirus B-3. CB-3 virus is not neutralized by any of the A-H pools since specific antiserum does not appear in the pools. If the field isolate is not neutralized by any of the A-H pools, 10^{3.0} TCD₅₀ of the virus should be tested against 50 units of CB-3 antiserum. The 2 ml vial should be reconstituted in 2 ml of distilled water. An appropriate quantity of the rehydrated serum should then be diluted 1:80 in Melnick's medium B to obtain 50 antibody units per 0.1 ml. (ii) Echovirus 11. Echovirus 11 antiserum appears only in pool E but unless large doses of Ell virus are employed, the virus may be neutralized by the heterotypic antibody of pools A and G, which is mainly associated with the E5 serum present in these two pools, as well as in pool E. Therefore, if neutralization by pools AEG occurs, special tests are required

to determine whether the isolate is either Ell virus or E5 virus. $10^{3.0}~{\rm TCD}_{50}$ of the virus should be tested against 50 units of Ell serum. The 2 ml vial should be reconstituted in 2 ml of distilled water. An appropriate quantity of the rehydrated serum should then be diluted 1:40 in Melnick's medium B to obtain 50 antibody units per 0.1 ml. If neutralization occurs, the virus is Ell; if not, the virus is E5. (iii) Echovirus 12. The specific El2 antiserum appears in pools C and H; however, there is also a strong heterotypic neutralization of E12 virus by pools A, E, and G and a weak neutralization by pool F. Therefore, test results on a isolate neutralized by C and H and any one or more of pools A, E, F and G indicate the need for application of a special test to identify the virus unequivocally as echovirus 12. $10^{3.0}$ TCD₅₀ should be tested against 50 units of El2 serum. The 2 ml vial should be reconstituted in 2 ml of distilled water. An appropriate quantity of the rehydrated serum should then be diluted 1:520 in Melnick's medium B to obtain 50 antibody units per 0.1 ml. Neutralization identifies the virus as El2.

b. The Echovirus 22/23 Complex. Echovirus 22 antiserum appears in pools E and H; echovirus 23 serum in pools G and H. Unless large doses of virus are employed, the two viruses are not clearly distinguished, but may be neutralized by E, G and H pools. Titration of the isolate against both E22 and E23 antisera is required for positive identification of the subtype. Also heterotypic neutralization

of small doses of E22 virus may occur with pool B. However, this need not confuse identification since none of the other enteroviruses can be identified by this combination (BEH). The homologous antibody titer assigned to the E22 equine serum is 1:13,200 per 0.1 ml; that of the E23 serum is 1:16,000

The homologous antibody titer of CB3 serum is 1:4000 per 0.1 ml; that of Ell serum is 1:2000; and that of El2 serum is 1:26,000.

IDENTIFICATION TABLE

Pool Neutralization a	Virus Identity	Pool Neutralization	Virus Identity
A	E15	D	E25
AB	CA7	DE	E13
AC	CB1	DF	E14
AD	E33	DG	E16
AE	CB4	DH	Р3
AF	E7	DEH	E32
AG	E4	E	E11 <u>b</u>
AH	El	EF	E18
ACF	E29	EG	E17
AEG	E5 <u>b</u>	EH	E22 e
· В	E21	F	E27
ВС	E2	FG	E20
BD	CB2	FH	CB6
BE .	P2	G	E31 /
BF	E19	GH	E 23 ^e
BG(C)	CA9 C	Н	CA16
ВН	E3		
BDF	E26		
BFH	E9		
С	E24		
CD	E6		
CE	CB5		
CF	P1 .		
СН	E12 <u>d</u>		
CEG	E30		

Footnotes for Identification Table:

- a CB3 antiserum, because of labelling error, does not appear in any of the pools.

 No neutralization of an enterovirus isolate by pools A-H strongly suggests that the isolate may be CB3 virus. For identification of CB3, see Special Tests above.
- b Pools AEG contain E5 antiserum, which may give a heterotypic neutralization of E11 virus. To distinguish between E5 and E11 viruses, see Special Tests above.
- Strong neutralization by B and G pools and weak neutralization (or no neutralization) by pool C identifies the virus as CA9. The C pool inadvertently, because of a labelling error, contains 10 antibody units against CA9 virus.
- Heterotypic neutralization of E12 by pools A, E, F and G may occur. For positive identification of E12, see Special Tests above.
- e Neutralization by pools E, G and H indicates the isolate may be either E22 or E23 virus. See Special Tests concerning the E22/23 complex.

PROTOCOL 1 IDENTIFICATION OF A FIELD STRAIN OF COXSACKIEVIRUS B5

The 10^{-1} dilution of virus was used in the test.

The virus was completely neutralized by pools C and E, the two pools containing CB5 antiserum.

Pools	Observation for CPE * on day					
	2	3	4	5	6	7
A	4,4					
В	4,4					
c ·	0,0	0,0	0,0	0,0	0,0	0,0
D	4,4					
E	0,0	0,0	0,0	0,0	0,0	0,0
F	4,4					
G	4,4					
Н	4,4					
Virus controls					,	
10-1	4,4,4,4	:				
10 ⁻²	2,2,2,1	4,4,4,4				
10 ⁻³	2,0,0,0	4,4,3,3	4,4,4,4			
10-4	0,0,0,0	2,2,0,0	4,4,0,0	4,4,0,0	4,4,0,0	4,4,0,0
10 ⁻⁵	0,0,0,0	0,0,0,0	0,0,0,0	0,0,0,0	0,0,0,0	0,0,0,0

^{*}Figures represent degree of CPE in each inoculated tube.

O = no CPE; 1 = 1+ or 25% CPE; 2 = 2+ or 50% CPE; etc.

PROTOCOL 2 IDENTIFICATION OF A FIELD STRAIN OF ECHOVIRUS 14

The $10^{-2.6}$ virus dilution was used in the test.

The virus was completely neutralized on day 5 by pools D and F which contained E14 antiserum, but breakthrough was noticed on days 6 and 7.

Pools	Observation for CPE * on day					
	2	3	4	5	6	7
A	0,0	2,2	4,4			
В	1,1	3,3	4,4			
С	1,1	3,3	4,4			
_D	0,0	0,0	0,0	0,0	0,0	1,1
E	1,1	3,3	4,4	·		
F	0,0	0,0	0,0	0,0	1,0	2,0
G	1,1	3,3	4,4			
H·	0,0	2,3	4,4			
Controls						
10 -2.6	2,2,2,2	3,3,3,3	4,4,4,4	,		
10 ^{-3.6}	0,0,0,2	1,2,1,3	4,4,4,4			
10-4.6	0,0,0,1	0,0,0,3	0,0,0,4	1,0,0,4	2,1,0,4	4,2,0,4
10 ^{-5.6}	0,0,0,0	0,3,0,0	0,0,0,0	0,0,0,0	0,0,0,0	0,0,0,0

^{*}Figures represent degree of CPE in each inoculated tube.

 $^{0 = \}text{no CPE}$; 1 = 1 + or 25% CPE; 2 = 2 + or 50% CPE; etc.

REFERENCES

- 1. Melnick J.L. and Hampil, B. (1965). WHO collaborative studies on enterovirus reference antisera. Bull. Wld. Hlth. Org. 33: 761-772.
- 2. Hampil, B. and Melnick, J.L. (1968). WHO collaborative studies on enterovirus reference antisera. Second report. Bull. Wld. Hlth. Org. 38: 577-593.
- 3. Melnick, J.L. and Hampil, B. (1970). WHO collaborative studies on enterovirus reference antisera. Third report. Bull. Wld. Hlth. Org. 42: 847-863.
- 4. Lim, K.A. and Benyesh-Melnick, M. (1960). Typing of viruses by combinations of antiserum pools. Application to typing of enteroviruses (Coxsackie and ECHO).
 J. Immunol. 84: 309-317.
- Melnick, J.L. and Wenner, H.A. (1969). Enteroviruses. Chapter 17, pp. 529-602, in "Diagnostic Procedures for Viral and Rickettsial Infections" (eds. E.H. Lennette and N.J. Schmidt), 4th Edition, Am. Publ. Health Assn., New York.
- Schmidt, N.J., Melnick, J.L., Wenner, H.A., Ho, H.H. and Burkhardt, M.A.
 (1971). Evaluation of enterovirus immune horse serum pools for identification of virus field strains. Bull. Wld. Hlth. Org. 45:317-330.
- 7. Wallis, C., and Melnick, J.L. (1967). Virus aggregation as the cause of the non-neutralizable persistent fraction. J. Virol. 1: 478-488, 1967.

MELNICK'S MEDIUM B

Component	Gram/Liter
NaCl	6.80
KCI	0.40
NaH ₂ PO ₄ · H ₂ O	0.14
CaCl ₂ (anhyd.)	0.20
MgS04 · 7H20	0.20
Glucose	1.00
Lactalbumin hydrolysate	5.00
Phenol red	0.01
NaHCO ₃	2.20

Ref.: Melnick, J.L., Ann. N.Y. Acad. Sci., 61:755 (1955).

PARASITE ASSAY

PARASITE SAMPLE COLLECTION

The off-site laboratory utilized a modified version of EPA's 1978 method for detecting <u>Giardia</u> and other parasites in water. The EPA collection device consisted of the following:

- inlet hose
- plastic cartridge filter housing
- orlon fiber filter wound on a polypropylene support (nominal porosity of 5 µm)
- effluent hose
- water meter

SAMPLE PRESERVATION

Generally, 100 to 500 gallons were concentrated depending upon the purity of the water. Flow rates of 11.4 L/min at line pressures of 15 to 70 psi were maintained during sample collection. After sampling, each filter was placed in polypropylene zip-lock bag and stored at 4°C. A ten percent Formalin fixative solution was added to the bag containing the filter. Preserved filters in the bags were shipped to the off-site laboratory for analysis.

SAMPLE PROCESSING

The filter was aseptically removed from the zip-lock bag and placed into a tray. A razor blade was used to cut an end strand from the filter such that the entire filter could be unwound from the core. The resulting filter mat was kneaded in 250 ml distilled water until the fibers appeared clean. The fibers were squeezed to remove as much water as possible, and the water was then centrifuged for three minutes at 450 g. The resulting sample pellet was washed three times by resuspending the pellet in 30 ml distilled water. The material was resuspended a final time and centrifuged for five minutes at 450 g. The supernatant was aspirated until approximately 50 ml remained. This material was transferred to a 50 ml conical tube and centrifuged for three to five minutes at 350 g. As much supernatant as possible was removed without disturbing the pellet.

PARASITE CONCENTRATION

To concentrate the sample material, the zinc sulfate (ZnSO₄) flotation technique was utilized to separate the parasitic components from excess debris through differences in specific gravity. The ZnSO₄ solution had a specific gravity of 1.18-1.20. Protozoan cysts and certain helminth eggs float to the top and are recovered in the surface film while the debris settles to the bottom of the tube. The pellet was vortexed with 5 to 10 ml ZnSO₄ and then transferred to a 15 ml conical tube. The tube was slowly filled to the brim with ZnSO₄ without allowing any runover. It was centrifuged for five minutes at 300 g without braking. The tube was carefully removed from the centrifuge without any agitation or vibration.

PARASITE STAINING

A drop of either Lugol's or D'Antonio's Iodine stain was placed on a clean, grease-free microscope slide. A wire loop (diameter of 5 to 7 mm) was used to remove one drop from the center of the tube's surface film and placed on the prepared slide. A clean No. 1 coverslip (22 x 22 mm) was placed upon the stained material.

PARASITE IDENTIFICATION

135501 TOSOCIOLO TOSOCIOLO TOSOCIOLO SISSEMINO SISSEMINO SISSEMINISTA INCIDENTI

THE RESIDENCE OF THE STATE OF T

The stained slide was examined microscopically under 20x and oil immersion. The criteria listed in Table A.1-1, especially parasite size and characteristic morphology, were used for identifications. EPA has established strict guidelines in order to report positive identifications. In most cases the size and observation of one or more characteristic morphological features, (i.e., nuclei, organelles, terminal knobs) were required for positive indentification.

Larval-form worms were observed in project samples. The method does not allow for identifying the worms as to whether they are of human or animal origin. Therefore, they were reported as larval-form worms.

TABLE A.1-1 PARASITE IDENTIFICATION CRITERIA

Species	Size	Shape	Number of <u>Nuclei</u>	Other
Giardia: cyst	8-19 µm Usual range: 11-14 µm Width: 7-10 µm	Ovoid	4	Longitudinal fibers in cyst
Entamoeba histolytica:	10-20 μm Usual range: 12-15 μm	Spherical	Mature cyst: 4 Immature: 1-2	Chromatoidal bodies and karyosome present
Acantha- moeba:	10 µm	Spherical	4	Culture for positive confirmation
Naegleria gruberi: cyst	10 μm	Spherical	1	
Ascaris: eggs	One-cell stage 45-75 µm by 35-50 µm	-	NA	Thick cortical shell
	Infertile 85-95 µm by 43-47 µm			
Hookworm: eggs	56-75 μm by 36-40 μm	_	NA	Thin-shelled, embryonated at times
Trichuris trichiura: eggs	50-54 μm by 20-23 μm	_	NA	Thin-shelled, mucoid plugs at each end, unembryonated

ACCOUNT CONTROL CONTRO

AMES SALMONELLA/MAMMALIAN-MICROSOME TEST METHOD FOR DETECTING MUTAGENS IN WATER AND WASTEWATER CONCENTRATES

PRINCIPLES OF THE AMES TEST

The Ames test is a rapid and simple bacterial bioassay for detecting chemical mutagens. Specially constructed mutants of Salmonella typhimurium are exposed to chemicals in an agar medium containing a minimum amount of histidine. All the strains have a histidine requirement that allows for the detection of revertant bacteria, which regain the ability to manufacture their own histidine from a reverse mutation in the DNA of the histidine-requiring mutant.

The trace amount of histidine in the top agar allows all the bacteria on the plate to undergo several divisions; this growth is necessary in many cases for mutagenesis to occur. The slight background lawn that grows up also allows any inhibition of growth by the test compound to be seen. Further increases in the amount of histidine on the plate enhances mutagenesis, but also causes heavy growth of the background lawn that obscures the revertants. The appearance of colonies indicates true histidine revertants. Colonies formed on plates without the incorporation of the test compound or mixture are called spontaneous revertants.

Some chemical mutagens and carcinogens require metabolic activation in vivo. Mammalian metabolism is incorporated into the in vitro Ames test by adding mammalian liver homogenates directly to the petri plates. The liver homogenate contains microsomal enzymes which may activate or deactivate chemicals, producing greater or fewer revertants compared to the plates without the enzymes. In some cases, the addition of liver homogenate has no noticeable effect either way.

The Ames test is a useful tool to obtain information about the potential mutagenic/carcinogenic activity of uncharacterized compounds in complex mixtures, such as organic concentrates from water and wastewater.

The following method is essentially the same as the protocol by Ames, et al published in <u>Mutation Research</u>, 31:347-364 (1975).

SALMONELLA STRAINS

Two tester strains of S. typhimurium (TA98, TA100) were obtained from Dr. Bruce Ames at the University of California, Berkeley. The two strains were incubated for 24 hours at 37°C in Oxoid Nutrient Broth #2. Each culture was streaked on several Oxoid agar plates. Isolated colonies from these plates were used to inoculate broth cultures from which frozen permanent cultures were made by freezing 0.8 ml of the culture and 0.07 ml of dimethyl sulfoxide (DMSO) at -70°C. Several permanents were separately stored to generate a new series of frozen permanents. The use of the frozen culture in this way avoids exposing the bacteria to repetitive freezing and thawing thus avoiding the loss of the R-factor plasmid which could result in a drop in the rate of

spontaneous reversion and inability to obtain the expected number of revertants with standard mutagens.

Master plates were prepared in accordance with the <u>Supplement to the Methods Paper</u>, Ames, et al (1975). Master plates were a convenient means of storing the two cultures in a conventional refrigerator at 4°C. The plates were the source of innoculum for the overnight cultures used in the mutagenesis assays.

Cultures of TA98 and TA100 were grown up from the frozen permanents in Oxiod Nutrient Broth #2; incubation was at 37°C for a period of 24 hours. Some of each culture was transferred to the Master Plates using a sterile wire loop in 5 or 6 parallel streaks across the plate. Subsequently, the plates were incubated overnight at 37°C and stored at 4°C.

New Master Plates were made from frozen permanents. Sub-culturing new Master Plates from old Master Plates might result in loss of the rfa mutation or the R-factor. A frozen permanent was discarded after being used to generate a set of master plates.

QUALITY CONTROL CHECK FOR THE MUTATION INTEGRITY OF TESTER STRAINS

Salmonella strains were tested for their specific types of mutations. These checks were run when the strains were first received from Dr. Ames and repeated with each mutagenicity test series.

Histidine Requirement

MAKAKA SASASA MAKAMA MAKAMA

KKE TERSOON KEEKKES DEEDLES KEEKKES AND SON

0.1 ml of 0.5 mM biotin was spread on two minimal agar plates. 0.1 ml of 0.1 mM histidine was spread on one of the plates. Each plate was streaked from an overnight broth culture of the tester strains. Both the TA98 and TA100 strains should grow only on the plate with histidine and biotin added.

Deep Rough (rfa) Character/R-Factor

0.05 ml of the tester strain and 0.25 ml of 0.5 mM histidine/biotin were added to 2.5 ml of molten top agar at 45°C. The contents were mixed and poured slowly on Oxoid agar plates. After the top agar had solidified, a filter disc containing 100 ug/L of 1 mg/10 ml aqueous crystal violet was asceptically placed on the top agar. A filter paper disc containing 10 ug of ampicillin was asceptically placed on the top agar of a similarly prepared plate. Both plates (two for each tester strain) were inverted and incubated at 37°C for 48 hours.

Both the TA98 and TA100 strains should show a zone of growth inhibition around the crystal violet disc, indicating the presence of the rfa mutation. Both of these strains (containing the R factor) are not inhibited by ampicillin.

Spontaneous Reversion

Minimal glucose agar plates and top agar were prepared as described by Ames, et al., (1975). 0.05 ml of the tester strain and 0.150 ml of dimethyl sulfoxide were added and mixed into 2.5 ml of top agar, poured on to minimal agar plates, incubated in an inverted position for 48 hours at 37°C and then counted. Between two and four spontaneous revertant control plates were prepared during each assay. Similarly, plates were prepared using 0.05 ml of the tester strain, 0.150 ml of dimethyl sulfoxide and 0.500 ml of S-9 mix

Genotype of Revertant Colonies

The genotypes of revertant colonies were checked by transferring revertant colonies on to minimal glucose agar plates containing 0.1 ml of 0.5 mM biotin but no histidine. True revertants to histidine prototrophy will grow on the minimal agar plates.

PREPARATION OF MEDIA, S-9 REACTION MIXTURE AND TESTER STRAINS

Base Medium

MERSON MARKEL SHEETS HENDER MARKET

PRODUCTION ACCESSES RESISTANCE OF TAXABLE CONTRACTOR OF

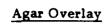
50X VB Salts. The ingredients were added in the order indicated and each salt was allowed to dissolve before adding the next one. When the salts were completely dissolved, the solution was autoclaved at 121°C for fifteen minutes.

50X Vogel-Bonner Salts

1.	Warm Distilled water (45°C)	670 ml
2.	MgSO ₄ ·7H ₂	10 g
3.	Citric acid, monohydrate	100 g
4.	K2HPO4	500 g
5.	NaHNH4PO4	175 g

40 Percent Glucose. 200 g of glucose was dissolved in 345 ml of distilled water and 100 ml and 75 ml aliquots were dipensed in prescriptions bottles, and autoclaved at 121°C for fifteen minutes.

Minimal Glucose Plates. 20 ml of 50X VB salts and 50 ml of 40 percent glucose were added to 1 L of molten agar (15 g Difco Agar, Purified/L distilled water, autoclaved at 121°C for 15 min). The agar was mixed, cooled to 45°C, and then poured into petri dishes (25 ml/dish). The plates can be stored at room temperature for several weeks but were routinely used in the assay within 24 hours.



Top agar (0.6 percent Agar-Purified, Difco and 0.5 percent NaCl) was prepared on the day of the assay. After autoclaving at 121°C for 15 minutes, the agar was placed in a water bath at 45°C and allowed to cool. 1 ml of 0.4 mM biotin and 0.5 mM histidine was added per 10 ml of top agar and mixed thoroughly by gentle swirling. The top agar (2.4 ml) was dispensed into sterile 13 x 100 mm culture tubes in a heating block at 45°C.

Histidine-Biotin Mix

L-Histidine HCl (Monohydrate) (mw 191.7)	0.09585 g (0.5 mM)
D-Biotin (mw 224.3)	0.1222 g (0.5 mM)
H ₂ 0	1,000 ml

The mixture was prepared in a volumetric flask and stirred for an hour. It was then sterilized by filtration through a 0.45 µm pore size filter and stored at 4°C.

S-9 Rat Liver Reaction Mixture

The rat liver S-9 homogenate (Arochlor 1254 induced) was purchased from Litton Bionetics. The S-9 mix was prepared as follows:

Reagent	Concentration	Volume
MgCl ₂	0.4 M	0.9 ml
KC1	1.65 M	0.9 ml
D-Glucose-6-Phosphate	1.0 M	0.225 ml
B-Nicotinamide Adenine Dinucleotide Phosphate (NADP)	0.1 M	1.8 ml
Buffer Mix	0.2 M	22.5 ml
Rat Liver Homogenate		5.0 ml
Sterile Distilled Water		18.7 ml
		50.0 ml

- 1. MgCl₂ (0.4 M): 8.132 g of MgCl₂ was dissolved in 100 ml of distilled water, autoclaved for 15 minutes at 121°C and stored at 4°C.
- 2. KCl (1.65 M): 12.30 g of KCl was dissolved in 100 ml of distilled water, autoclaved for 15 minutes at 121°C and stored at 4°C.
- 3. D-Glucose-6-Phosphate (G-6-P) (1.0 M): 2.821 g of G-6-P (Monosodium salt, Sigma Chemical Company, No. G-7879) was dissolved in 10.0 ml of distilled water. It was sterilized by filtering through a Gelman Acrodisc R filter, 0.2 µm pore size and then dispensed into sterile 2 ml vials and stored at -20°C.
- 4. NADP (0.1 M): 0.765 g of NADP (Monosodium salt, Sigma Chemical Company, No. N-0505) dissolved in 10.0 ml of distilled water. It was sterilized by filtering through a Gelman Acrodisc R filter, 0.2 µm pore size, and then dispensed into sterile 6 ml vials, and stored at -20°C.

5. Buffer Mix

- a. Na₂HPO₄·7H₂O, dibasic (0.2 M): 5.36 g and Na₂HPO₄ was dissolved in 100 ml distilled water
- b. NaH₂PO₄·H₂O, monobasic (0.2 M): 2.76 NaH₂PO₄·H₂O was dissolved in 100 ml distilled water.

The buffer was prepared by mixing the two salts as follows:

82 ml Na₂HPO₄·7H₂O (0.2 M) 18 ml NaH₂PO₄·H₂O (0.2 M) 100 ml

Test Strain Suspension

The test culture was prepared by aseptically innoculating 25 ml of Oxoid Nutrient Broth; the inoculum source was a Master Plate as described above. The culture was incubated for eighteen hours at 37°C with slight agitation. Approximate bacterial density for both the TA98 and TA100 strains was 1×10^9 organisms per ml.

PLATE INCORPORATION ASSAY

Assay

The following were added (in order) to 2.5 ml of molten top agar at 45°C (tubes in heating block): test sample (dissolved in DMSO) sulfoxide) not exceeding 0.250 ml, 0.05 ml of the bacterial suspension, and 0.50 ml S-9 mix (when included). The contents were immediately mixed by rotating the tube between palms for several seconds and overlayed on a minimal glucose agar plate. Plates were inverted and incubated 48 hours at 37°C in a dark incubator, Duplicate plates were made for each concentration.

Spontaneous reversion was determined by adding 0.05 ml of the bacterial suspension and 0.150 ml of DMSO to the top agar. To determine the effects of enzyme activation, 0.500 ml of S-9 mix, in addition to 0.05 ml of the bacterial suspension and 0.150 ml of DMSO, was incorporated into the top agar. Spontaneous reversion rates, with and without S-9 activation, were based on counts obtained from duplicate, triplicate or quadruplicate plates.

Sterility controls were conducted by adding DMSO, S-9 mix and biotin-histidine to separate plates. In addition, top agar alone was poured on duplicate minimal agar plates to check for sterility of the top agar.

Scoring Revertants

After 48 hours incubation, the revertant colonies were counted either manually (Quebec Colony Counter) or with an automatic counter (Artek Counter, Model 880). All the plates inoculated with the TA98 strain were counted manually while all the plates inoculated with the TA100 strain were counted automatically. Approximately ten percent of the plates counted on the automatic counter were cross-checked manually to verify accuracy.



The presence of a light background lawn of growth (due to the trace amount of histidine added to the top agar) was confirmed by viewing plates under a microscope or against room light.

Safety Procedures

- 1. Aseptic technique was used when handling the bacterial strains. Pipetting was done by bulb or automatic pipette, no mouth pipetting.
- 2. All work done with the known mutagens was done under the hood. Lab coats and disposable gloves were worn.
- 3. Test plates were collected in autoclavable bags and autoclaved before disposal.
- 4. Liquid mutagenic material was collected in a glass bottle marked "Toxic Waste" and disposed of with toxic chemicals.
- 5. The work area was washed with a disinfectant before and after use. Hands were washed after doing lab work.

TESTING FOR IN VITRO TRANSFORMATION IN THE C3H/10T1/2 MOUSE EMBRYO FIBROBLAST SYSTEM

BACKGROUND

Many short-term assays utilizing different test organisms and test endpoints have been developed for toxicological testing. However, no single assay is capable of detecting all carcinogens and many investigators favor using a battery of short-term tests to gather toxicological data. The use of mammalian cells, though more costly and time-consuming than bacterial systems, has the advantage of being more closely related to human cells with respect to its DNA and metabolic activities.

Over the past ten years, a number of mammalian cell culture systems have been developed in which non-malignant cells have been transformed (changed) following exposure in vitro to chemical carcinogens. Although originally developed to study the events leading to carcinogenesis, these tests show great promise as rapid tests for screening potential carcinogens. Reproducible quantitative systems utilizing cell strains and cell lines have correctly identified many carcinogens. When the transformed cells are injected into suitable hosts a high correlation with tumorigenicity is observed.

The mammalian cell transformation assay used in this project was developed by Dr. Charles Heidelberger and his co-workers. The system utilizes a fibroblastic cell line, C3H/10T1/2, derived from C3H mice. The cell line has an extremely low rate of spontaneous transformation as well as a very flat morphology, making it relatively easy to score morphologically transformed foci. In this system, morphologic cell transformation is characterized by the cells' loss of density-dependent inhibition, resulting in the formation of colonies in which the cells have piled up.

The C3H/10T1/2 cell line has been transformed with polycyclic aromatic hydrocarbons with a quantitative dose response. In addition, the cell line was found to be transformable with chemically reactive carcinogens such as MNNG and N-Ac-AAF, several cancer therapeutic agents, tobacco smoke condensates and hair dyes.

The assay involves exposing actively growing cell monolayers to the test material for 48 hours. The cells are then rinsed and maintained at 37°C in five percent CO₂ for six weeks at which time the cells are stained with giemsa and examined under a dissecting microscope for morphologically transformed foci. Three classes of foci can be identified. Type I is a focus composed of tightly packed cells. Type II foci show massive piling up into opaque multilayers in which criss-crossing is not pronounced. Type III foci are multi-layered criss-crossed arrays of densely stained cells. The transformation frequency is calculated as the percentage of cells that give rise to transformed foci, corrected for the fraction of cells surviving.



General Lab Procedures

All manipulations were carried out aseptically in a laminar flow hood. No antibiotics were used in any of the cultures, so a clean environment must be maintained. Access to the "clean room" was limited to individuals who must work in there. A tacky mat was positioned just inside the door to trap dust carried in on shoes or cart wheels. Incoming air was HEPA-filtered and the air was routinely monitored for airborne microbes. A known volume of air was drawn through an open filter holder containing either a 0.8 or 0.45 µm membrane filter. The filter was then removed and incubated on Sabouraud dextrose or trypticase soy agar plates and checked for growth of mold or bacteria.

The CO₂ content of the incubator was monitored regularly with a fyrite tester. The chambers of the incubator were kept clean and disinfected after each assay. The surfaces were cleaned with mild detergent, thoroughly rinsed with deionized water and then wiped with seventy percent ethyl alcohol. Any spills were wiped up immediately to prevent the growth of microorganisms in the incubator, and opening of incubator doors was kept to a minimum to prevent fungal spores from being drawn into the chamber.

General Cell Maintenance Procedures

Stock cultures were grown in plastic tissue culture flasks (Corning, 25 or 75 cm²). Complete medium consisted of Eagles Basal Medium (BME) (see Table A.1-2) with L-Glutamine, without NaHCO₃ (GIBCO) plus ten percent heatinactivated fetal calf serum. Cells were incubated in a humidified, five percent CO₂ environment at 37°C. Stock cultures were refed on day nine and passaged on day ten.

Cell Transfer Technique

Ten day old monolayers were rinsed with Ca⁺⁺/Mg⁺⁺-free phosphate buffered saline (see Table A.1-3) and dispersed with 0.1 percent trypsin. When cells reached the single-cell stage, fresh medium containing serum was added to stop the action of trypsin. The cells were centrifuged and resuspended in fresh medium. An aliquot was counted with a hemacytometer and the appropriate number of cells seeded in flasks or petri dishes.

For stock cells, T-flasks were seeded with 5×10^4 cells. For transformation assays each 60 mm petri dish was seeded with 2000 cells. Five 60 mm dishes per dose were seeded with 200 cells to determine plating efficiency.



STATES OF STATES STATES STATES STATES

TABLE A.1-2 EAGLES BASAL MEDIUM

Component	mg/L_
CoClo	200.00
CaCl ₂ KCl	400.00
MgSO ₄	97.67
MgSO ₄ ·7H ₂ O	6800.00
NaH2PO4·H2O	140.00
Glucose	1000.00
Phenol Red	10.00
L-arginine HCl	21.00
L-cystine-2HCl	15.65
L-Glutamine	292.00
L-Histidine	8.00
L-Isoleucine	26.00
L-Leucine	26.00
L-Lysine HCl	36.47
L-Methionine	7.50
L-Phenylalanine	16.50
L-Threonine	24.00
L-Tryptophan	4.00
L-Tyrosine	26.00
Biotin	1.00
D-Ca pantothenate	1.00
Choline chloride	1.00
Folic acid	1.00
i-inositol	2.00
Nicotinamide	1.00
Pyridoxal HCl	1.00
Riboflavin	0.10
Thiamine HCL	1.00
I mamme ucr	1.00

TABLE A.1-3 PHOSPHATE BUFFERED SALINE

Component	g/L
KCI	0.20
KH2PO4	0.20
NaCl	8.00
Na ₂ HPO ₄	1.15

QUALITY ASSURANCE

Media Sterility

Dry powdered BME was reconstituted in 950 ml of glass-distilled water and filtered by pressurization through a 0.22 µm cartridge filter into sterile bottles. From each batch of prepared media, one out of every ten bottles was tested for sterility. Each test bottle was incubated at 37°C under five percent CO₂ for ten days and then examined for growth.

Cell Line

No antibiotics were used in any of the cultures since they can mask chronic infections or cause the emergence of bacterial L-forms.

Each batch of fetal calf serum was tested for its ability to support growth of the cells, and maintain a monolayer for six weeks.

Positive and Negative Controls

Whenever a transformation assay was performed, 3-methylcholanthrene (MCA), a known carcinogen, and DMSO, a negative solvent blank were included in the assay.

PROCEDURES FOR THE ASSAY

Transformation Assay

ES VERPERO PERSONA L'ESCENT ANTORIN ACCESSO DECRETA SECURITA MANTEN ANTORIN ANTORINA DE PROPERTO DE PARTICIONA

Each sample was tested for transformation at three dose levels. For each dose level twenty dishes, with 2000 cells per dish, were treated. Each experiment also included positive and negative controls in the same number of dishes. Twenty-four hour cultures were exposed to the test material for 48 hours at which time the medium was removed, the cells were rinsed and fresh medium added. Medium was changed twice weekly until the cells reached confluence and weekly after that. Six weeks after treatment the cells were stained and examined for transformed foci.

Plating Efficiency

Plating efficiency was determined by exposing 200 cells (five dishes per concentration) to the test material. Conditions were the same as in the transformation assay. At the end of ten days, the cells were stained and the colonies counted to determine the survival fraction. The transformation frequency was defined as the percentage of cells giving rise to Type II and Type III foci, corrected for plating efficiency, i.e., number of foci per surviving cell.

ANALYSIS OF ORGANIC CARBON BY PHOTOCHEMICAL OXIDATION/ INFRARED ABSORPTION

DEFINITION

The analysis of organic carbon consists of 1) elimination of inorganic carbon interferences by acidification and purging, 2) oxidation of organic carbon to carbon dioxide in the presence of persulfate and ultraviolet light, and 3) measurement by infrared absorption of the carbon dioxide produced. Organic carbon can be measured over a concentration range of 0.05 to 2000 mg/L.

APPARATUS

Dohrmann-Xertex DC-80 automated laboratory total organic carbon analyzer consists of an auto sampling module, a reaction module, a detector/electronics module, and a printer module.

REAGENTS

- A. Organic-Free Water is generated by passing deionized distilled water through a stainless steel ultraviolet reaction chamber at 2 liters per half hour. This water is collected in a 2 liter amber bottle and tightly sealed.
- B. Potassium Hydrogen Phthalate ACS grade
- C. Phosphoric Acid ACS grade
- D. Potassium Persulfate ACS grade

PREPARATION OF STANDARDS AND REAGENTS

A. Stock Standard Solutions

- 2000 mg/L potassium hydrogen phthalate (KHP). Weigh accurately 2.1250 grams of potassium hydrogen phthalate into a small beaker. Quantitatively transfer to the 500 ml flask. Bring up to volume with reagent water. Add twenty drops of acid. Add teflon coated magnetic bar, and stir for approximately one hour.
- 2. 200 mg/L potassium hydrogen phthalate. After rinsing the designated 50 ml pipet with 2000 mg/L standard, transfer 50 ml of 2000 mg/L KHP to the 500 ml flask, bring up to volume with reagent water. Add twenty-five drops of phosphoric acid, stopper, and invert the flask several times.

B. Working Standard Solutions

- 1. A 10 mg/L KHP standard is prepared by dilution of 25 mls. of 200 mg/L stock standard in 475 mls of reagent water, and adding 25 drops of phosphoric acid.
- 2. 5, 3, 2 and 1 mg/L standards are prepared by pipeting 100, 60, 40 and 20 ml of 10 mg/L respectively into the appropriate 200 ml flasks bringing each up to volume with reagent H₂O and adding 10 drops of phosphoric acid. NOTE: A 0.5 mg/L standard is also prepared by dilution of 10 mls of 10 mg/L standard in 190 mls of reagent H₂O when a sample's organic carbon content is below 1 mg/L. If the organic carbon content of a sample exceeds 10 mg/L, a 25 mg/L standard is prepared by dilution of 25 mls of 200 mg/L standard with 10 drops of acid in 175 mls of water.
- Ultraviolet reactor reagent is prepared by dissolving approximately 40
 grams of potassium persuflate in one liter of water and adding 0.5 mls
 of phosphoric acid.

ANALYSIS

the testing resided the property to the property of the proper

A. Sample Preparation

- 1. After inverting the sample bottle several times, a small quantity of sample is removed from each sample bottle.
- 2. The bottle is resealed and vigorously agitated.
- The sample is then quickly decanted into clean, labeled pyrex test tubes.
- 4. Raw water samples are analyzed in triplicate.
- 5. Finished water samples are analyzed in duplicate.
- 6. If a sample contains considerable particulate matter, test tubes containing reagent water are placed after each replicate on the auto sampling tray.

B. Calibration and Analysis

- 1. Each day, after the instrument has stabilized, four replicates of the 10 mg/L standard are analyzed.
- 2. In the value on the printer for the first two replicates are outside of 10.0 ± 0.5 the instrument is recalibrated using the third and fourth replicates for the calibration.
- 3. Duplicates of the 5, 3, 2 and 1 mg/L standards and the water blank are analyzed.

- 4. From an examination of the printer values for these standards, the analyst determines if the instrument is functioning properly. If it is not the analyst "trouble shoots" the instrument before proceeding.
- 5. If the instrument is functioning properly, a set of samples is placed on the autosampler starting with finished drinking waters and proceeding to samples of higher organic carbon content.
- 6. After each set of samples a complete set of standards and the water blank are analyzed in duplicate.
- 7. After placement of the test tubes on the autosampler tray, the purging, introduction of sample and the integration of peak area is performed by the instrument.

CALCULATION

- A. The replicates for each standard sample, and the water blank are averaged.
- B. The mean of the water blank is subtracted from the mean of each standard.
- C. A correction factor (defined as the actual value of the standard divided by the mean of the instrument's values for that standard minus the mean of the water blank) is determined for each standard.
- D. By interpolation from this correction factor curve the analyst determines the appropriate correction factor for each sample.
- E. The mean of each sample is multiplied by a correction factor and a corrected organic carbon value in mg/L is reported.

QUALITY ASSURANCE

Quality assurance is maintained by the following analytical routine:

- A. Before a newly prepared stock standard is introduced into the analysis it is analyzed with the stock standard being used at that time. If the mean values for the two standards are not within three percent of one another the new standard is discarded and another is prepared.
- B. New working standards are prepared for each analysis.
- C. If there are working standards (10, 5, 3, 2 and 1 mg/L) remaining from the previous analysis, these are analyzed and compared to the standards prepared on that day.
- D. All samples are run in duplicate or triplicate.
- E. For each sample, if the values for the duplicates are not within 10 percent of one another additional replicates are analyzed.



- F. 5 percent of all samples are spiked and recoveries are determined.
- G. After manual review of the data, the analyst submits a report to the lab clerk. The lab clerk proof reads the report for accuracy after it is generated.
- H. The analyst proof reads the report and signs it when satisfied that the results reported are valid.
- I. The final report is submitted to the senior organic chemist for final review. As a final check, the values reported are compared to the typical concentration range expected for a particular water supply.
- J. The worksheets, chromatograms, quantitation reports, etc. are filed according to client by the lab clerk. The final report is duplicated and placed in the organics file report binder, also according to client.
- K. The final report is sent out.
- L. The remaining portion of the water sample is stored for one month at 4°C to allow reanalysis should there be any question of the results.



ANALYSIS OF ORGANIC HALOGEN IN POTABLE AND NON-POTABLE WATER BY CARBON ADSORPTION

DEFINITION

The analysis of organic halogen consists of four parts: isolation and concentration of the organics from water by carbon adsorption, elimination of inorganic interferences, pyrolysis of the carbon to convert to inorganic halide, and determination of the halide by microcoulometric titration. The technique shows excellent recoveries for most halogenated organic compounds with the exception of some chlorinated alcohols and acids. Organic halogen is determined over a concentration range of approximately 5 to 500 µg/L. Actual detection limits are dependent on the daily performance of the halogen analysis system.

APPARATUS

Dohrmann-Envirotech DX-20 Total Organic Halogen Analyzer. The DX-20 consists of two modules, an adsorption module and an analyzer module. The adsorption module includes gas flow controls, sample reservoirs, carbon columns, automatic sample flow shut-off for simultaneous processing of four water samples, and a nitrate wash channel. The analyzer module includes a temperature controlled purgeable organic halogen sparger, automatic boat inlet, high temperature pyrolysis furnace, and microcoloumeter.

REAGENTS

- A. Activated Carbon 100-200 mesh
- B. Cerafelt
- C. Organic free water
- D. Methyl alcohol, sodium sulfite, acetic acid, nitric acid, sulfuric acid, sodium chloride —ACS reagent grade
- E. Standards -2,4,6-trichlorophenol and 2,4-dichlorophenol.

PREPARATION OF STANDARDS AND REAGENTS

- A. Stock Standard Solutions
 - 2,4,6-trichlorophenol (10 μg organicly bound chloride per μl). Weigh accurately 1.854 grams 2,4,6-trichlorophenol into a 100 ml volumetric flask. Add methanol to volume.
 - 2. 2,4-dichlorophenol (10 µg organicly bound chloride per µl). Weigh accurately 2.2958 grams of 2,4-dichlorophenol into a 100 ml volumetric flask. Add methanol to volume.

- B. Working Standard Solutions 2,4,6-trichlorophenol (1 μg Cl/1 μl). Dilute 10 mls stock solution to 100 mls with methanol.
- C. Reagent Water Organic-free water is generated by passing laboratory D.I. water through a Barnstead Organic pure instrument.
- D. Inorganic Chloride Microcoulometric-Calibration Standard (1000 ppm chloride) Accurately weigh 0.1648 grams reagent grade sodium chloride into a clean ground glass stoppered 100 ml volumetric flask. Fill to volume with reagent water.
- E. Nitrate-Wash Solution (5000 mg nitrate/L) Transfer approximately 8.2 grams of potassium nitrate into a one liter ground glass stoppered flask. Add one liter of reagent water. Stir until dissolved.
- F. Sodium Sulfite Reducing Agent (0.1 molar) Measure approximately 12.6 grams of reagent grade sodium sulfite into a ground glass stoppered flask. Add one liter reagent water. Stir until dissolved.
- G. Microcoulometric Cell Electrolite (70 percent acetic acid in water) Place 700 ml of Acetic Acid (glacial) and 300 ml reagent water in a clean flask. Invert flask several times.
- H. Sulfuric Acid (50 percent solution) Place 50 mls of sulfuric acid and 50 mls of reagent water in a 500 ml erlenmeyer ground glass stoppered flask.

SAMPLE CONCENTRATION AND ANALYSIS

THE PROPERTY SECURITY WAS ARRESTED TO SECURITY WHEN THE SECURITY OF THE SECURI

- A. Sample Concentration by Carbon Adsorption:
 - 1. 40 mg of 10-200 mesh granular activated carbon is deposited in each glass adsorption column. Both ends of the columns are plugged with cerafelt.
 - 2. Two adsorption columns are placed in series in the adsorption column housings.
 - 3. The housings are attached to the sample reservoir outlet.
 - 4. After inverting the sample several times, an aliquot is immediately poured into the sample reservoir. Avoid aeration by gently pouring the sample into the reservoir. The reservoir is sealed and placed under 20 psi of pressure.
 - 5. Depending on the nature of the sample, a volume of 25, 50, or 100 ml is filtered through the adsorption columns into a class A volumetric flask. When the appropriate volume of sample has been filtered, the pressure in the reservoir is automatically released, and filtering ceases.

- 6. The column housings are removed from the reservoir outlet and the sample remaining in the reservoir is allowed to empty into a waste receptacle.
- 7. The process is repeated in the same sample reservoir to produce a duplicate adsorbed sample.
- 8. Using the best available, acidified laboratory water, the reservoir is filled and drained three times in order to thoroughly rinse the reservoir.

B. Elimination of Inorganic Interferences:

The housings containing the two adsorption columns are placed on the nitratewash reservoir outlet. Under 6 psi pressure, two ml of nitrate solution are filtered through the adsorption columns to remove inorganic halides.

C. Analysis of Organic Halides:

Sample Introduction:

- 1. The carbon and cerafelt are quantitatively ejected from the adsorption columns into the quartz boat located in the analyzer inlet tube of the AD-2 analyzer module.
- 2. The inlet tube is sealed and the carbon dioxide flow through the inlet tube, pyrolysis tube and microcoulometric titration cell is reestablished.
- 3. After the machine has stabilized (approximately one minute) the automated analysis is begun by activation of the start switch.

Pyrolysis:

- 1. The quartz boat advances to the 250°C vaporization section of the pyrolysis tube where vaporizable components of the sample are swept through the pyrolysis section in the carbon dioxide stream.
- 2. After two minutes, the boat automatically advances to the 800°C section of the pyrolysis tube and the gas stream switches from carbon dioxide to oxygen.
- 3. After six minutes in the pyrolysis section, the boat is automatically retracted to the stand-by position in the inlet tube.
- 4. Two minutes after retraction of the boat, the analysis is completed and the machine returns to the ready condition.



Titration:

- 1. Throughout the ten minute analysis, organic halogens in the gas stream and in the quartz boat are combusted and the hydrogen halide products pass in the gas stream to the titration cell.
- 2. Through a constriction in the bottom of the titration cell, the gas is bubbled through the acetic acid solution of the cell.
- 3. Halides are trapped in the solution where they interact with silver ions, also present in solution to produce a silver halide precipitate.
- 4. The change in the concentration of silver ions is detected by sensor electrodes, producing a change in the voltage signal.
- 5. This change in voltage relative to a memorized baseline voltage is integrated and calculated. The net value, expressed as µg of chloride, is displayed on the digital meter.
- 6. The value is manually recorded in the laboratory notebook with the sample identification number, the reservoir on which it was filtered, the volume filtered, and the order in which all samples were analyzed throughout the day.
- 7. If the net values for the duplicates are not within 10 percent of each other, a third replicate is analyzed.

CALCULATION

HAN THE THE TOTAL HANDS HANDS THE TOTAL HANDS

and the property of the proper

- A. Means and standard deviations for samples, standards, and blanks are determined.
- B. The sample identification, means, standard deviations, volumes filtered, and theoretical standard values are entered into a computer program that corrects for standard calibration, volume, and background.
- C. The total organic halogen expressed as µg of organically bound chloride per liter are calculated.
- D. The sample identification number, TOX value, and standard deviation are stored on the H.P. computer and a hard copy of this information is produced.

QUALITY ASSURANCE

Quality assurance is maintained by the following analytical routine:

- A. The analyzer module is run with the boat empty at the beginning of each day to remove all contamination accumulated since the previous analysis.
- B. The response of the titration cell is checked each day by injecting 10 µg of inorganic chloride directly into the titration cell solution. Injections are

- repeated and adjustment of the instrument made until two subsequent replicates within $\pm 0.20 \,\mu g$ of 10 μg of chloride are obtained.
- C. Triplicate 10 µg organic chloride instrument calibration standards (standards injected directly onto the carbon contained in the adsorption columns) are analyzed at the beginning of each day, and subsequently after each 10 sample analyses.
- D. Triplicate carbon blanks (carbon packed addsorption columns washed with nitrate-wash solution only) are analyzed at the beginning of each day, and subsequently one blank is analyzed after each 10 sample analyses.
- E. All samples are analyzed in duplicate. If the net values of the duplicates are not within ten percent of one another, a third replicate is analyzed.
- F. The titration cell is revitalized by rinsing with fresh cell solution after approximately every twenty analyses.
- G. The volume of sample filtered is adjusted for each site analyzed to produce adsorbed organic halogen content in the range of optimum function of the instrument.
- H. Every twentieth sample, the top and bottom adsorption columns from one sample adsorption are analyzed separately to determine if any organic halogen breakthrough is occurring.
- I. Every twentieth sample is spiked and recoveries are determined.
- J. The purity and adsorption capacity of each new batch of carbon purchased is assessed by analysis of seven replicates each of the reagent water, carbon blanks, instrument calibration standards, and adsorption efficiency standards (standards injected into reagent water then filtered).
- K. After manual review of the data, the analyst submits a report to be typed to the lab clerk. The lab clerk proof reads the report for accuracy after it is typed.
- L. The analyst proof reads the report and signs it when satisfied that the results reported are valid.
- M. The final report is submitted to the senior organic chemist for final review. As a final check, the values reported are compared to the typical concentration range expected for a particular water supply.
- N. The worksheets, strip chart data, quantitation reports, etc., are filed according to client by the lab clerk. The final report is duplicated and placed in the organics files, also according to client.
- O. The final report is sent out.

P. The remaining portion of the water sample is stored for one month at 4°C to allow reanalysis should there be any question of the results.

ANALYSIS OF TRIHALOMETHANES IN POTABLE AND NON-POTABLE WATER BY LIQUID-LIQUID EXTRACTION

DEFINITION

The analysis of trihalomethanes consists of two parts: concentration by liquid-liquid extraction (LLE) and analysis by gas chromatography (GC). Four trihalomethanes — chloroform, dichlorobromomethane, dibromochloromethane, and bromoform — are determined over a concentration range of approximately 0.1 to 2000 µg/L. Actual detection limits are dependent on the daily performance of the chromatographic system.

APPARATUS

The Varian 4600 gas chromatograph is a modular, dual column, dual detection instrument capable of isothermal or temperature programmed operation with packed or capillary columns and a variety of detectors.

REAGENTS

- A. Organic-free water is generated by passing deionized distilled water through a Barnstead Organic unit. This water is collected in a narrow-mouthed 1 liter bottle and boiled for 15 minutes. While hot, contaminant-free nitrogen is bubbled through the water at 50 ml/minute continuously.
- B. Methyl alcohol ACS reagent grade
- C. Sodium thiosulfate ACS reagent grade
- D. Standards:
 - 1. Bromoform ACS grade
 - 2. Bromodichloromethane 97 percent ACS grade
 - 3. Chlorodibromomethane ACS grade
 - 4. Chloroform ACS grade
 - 5. Dibromopropane (I.S.) ACS grade
- E. Extractant pesticide grade pentane

STANDARD PREPARATION

- A. Fill a 50 ml glass stoppered volumetric flask with approximately 49 ml of methyl alcohol. Let stand for a few minutes. Stopper the flask and weigh to +0.1 mg.
- B. Add 3 to 4 drops of standard to the flask immediately with syringe or pasteur pipet and reweigh.

NOTE: The standard drops should fall directly into the alcohol without contacting the flask neck.

- C. Dilute to volume with methanol, stopper tightly and mix by inverting the flask 2 to 3 times.
- D. Calculate the concentration in µg per µl from the net gain in weight.
- E. Transfer 1 ml with a pipet into a 2 ml vial. Tightly cap, label, and store the solution at -20°C.

INTERNAL STANDARD

- A. Prepare a standard of dibromopropane in methanol as detailed above.
- B. From the calculated concentration determine the volume necessary to prepare a 1,720 µg/L solution. (This concentration is equivalent to 75 µg/L of water at the concentration ratio used in this procedure.)
- C. Using a syringe, inject this volume of concentrated dibromopropane in one liter of pesticide grade pentane. Fill to volume, stopper the flask and invert 2 to 3 times to mix the solution.
- D. Transfer and store at -20°C in 500 ml amber bottles with teflon seals.

EXTRACTION AND ANALYSIS

A. Aquecus standard solution for calibration

NOTE: Prior to preparation of standards check water blank on GC for impurities.

- 1. Prepare four (4) clean 500 ml glass stoppered volumetric flasks and two (2) 1 liter volumetric flasks, the automatic pipettor for phosphate buffer, and the automatic pipetor for the pentane and dibromopropane (internal standard) solution.
- 2. Fill the first 500 ml glass stoppered volumetric flask with approximately 500 ml organic-free water and label 300 ppb.
- 3. Inject 50 μ (the exact amount will depend on the number of μ g/ μ l) stock standard solution directly into the organic-free water.
- 4. Stopper the flask immediately and slowly invert 2 to 3 times to mix the solution. This solution will give a concentration of 300 parts per billion.
- 5. Immediately transfer the standard to 125 ml amber bottles and seal without headspace.

- 6. Inject 25 µl stock standard solution directly into the second 500 ml volumetric flask to obtain a 150 ppb standard.
- 7. Inject 17 µl stock standard solution directly into the next 500 ml volumetric flask to obtain a 100 ppb standard.
- 8. Inject 10 µl stock standard solution directly into the last 500 ml volumetric flask to obtain a 60 ppb standard.
- 9. Inject 10 µl of stock standard solution into the first 1 liter volumetric flask to obtain a 30 ppb standard.
- 10. Remove 50 mls of organic-free water from the remaining 1 liter flask and prepare the 5 ppb standard by pipetting 50 mls of 100 ppb standard into this liter flask.

B. Blank

- 1. The blank is the organic-free water used to prepare the standard.
- 2. Another blank consists of the extraction of organic-free water containing the buffer solution at the correct concentrations.
- 3. All blanks should be poured into labeled bottles to overflow and sealed with TFE teflon septa and screw caps. The blanks should then be checked for air bubbles as with the standards and samples.

C. Extraction Procedure

NOTE: Prior to extracting any sample, test the pentane on the GC for impurities.

- 1. Each 125 ml bottle containing standards and samples is opened, and 10 mls of water is removed using a pipettor with a disposable tip. 0.5 mls of phosphate buffer and 5 mls of pentane containing interval standard are added.
- 2. Bottles are then capped and left inverted on a level surface until it is ready to be placed on the shaker.
- 3. Shake the sample on a gyrorotatory platform shaker for 25 minutes at 400 rpm, then place sample on level surface and keep it standing in inverted position until extract collection.
- 4. Collect the extract with a disposable pasteur pipet and transfer into two 2 ml vials. Immediately seal the vials with a TFE septum and screw caps on tightly. Label each vial.
- 5. Put one labelled vial into the carousel of the autosampler of the 4600 gas chromatograph for analysis. Store the second vial at -20°C for repeat analysis if required.



CHROMATOGRAPHIC ANALYSIS

A. The THM's are analyzed with the following instrument conditions:

Detector: Ni⁶³ electron capture
Carrier Gas: Ultrapure N₂ @ 80 PSI

Injector Pressure:

Split flow:

Make-up flow:

Column flow:

Injector temp.:

Detector temp.:

15 PSI

30 ml/min

30 ml/min

220°C

300°C

Column temp.: 450 Isothermal

Autosampler injection volume: 2 µl

B. The column utilized is a fused silica open tubular column 0.25 mm x 30 M coated with SE-54. The trihalomethanes give the following retention times under these conditions:

CHCl ₃	4.2		
DCBM	5.4		
DBCM	7.8		
DPB (I.S.)	10.7		
CHBr ₃	12.6		

CALCULATION

With the information developed from the 1, 10, 50, and 100 PPB standards develop a calibration curve for each of the trihalomethanes. The calibration curve plots THM concentration versus integrated ion area counts. Ion counts are normalized with the internal standard according to the following calculation:

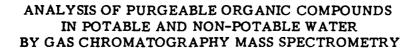
Normalized THM Ion Counts = (100,000) x (THM Ion Counts)
Internal Standard Ion Counts

QUALITY ASSURANCE

Analytical quality is assured by the following daily routine:

- A. Analyze extractant for impurities.
- B. Analyze blank water for impurities.
- C. Field blanks are recommended and analyzed as samples.
- D. Prepare and analyze standards at 5, 25, and 50 ppb in triplicate. Standards at 100, 150 and 300 are also routinely prepared and are analyzed when necessary.

- E. Develop calibration curves for each THM based on the average of the triplicate standards.
- F. Test precision by analyzing each twentieth sample of the day in duplicate. If standard deviation is >10 percent, troubleshoot problems before continuing.
- G. Spike each twentieth sample with the four THM's at between 10-50 ppb, check for accuracy. If accuracy is not ≤10 percent troubleshoot problems before continuing.
- H. Standards are included (approximately one every five samples) throughout the autosampling series.
- I. Prepare a written report of sample THM concentrations and have it typed by the lab clerk. The lab clerk proofs the report for accuracy in typing and addition of Total THM's. The analyst signs the report on the bottom when satisfied that the results reported are valid.
- J. All worksheets, chromatograms, and calibration curves are clipped to the final THM report and submitted to the chief organic chemist for final review. This review includes a spotcheck of the chromatograms to ensure proper resolution and quantification. As a final check the values reported are compared to the typical concentration range expected for the water supply and the CHCl3:DCBM:DBCM:CHBr3 ratio is reviewed.
- K. The worksheets, chromatograms, integrator values, and calibration curves are filed according to lab number by the lab clerk. The final report is duplicated and placed in the organic department's final report binder, according to lab number.
- L. The final report is sent to the client.
- M. The duplicate THM extract is stored at -20°C for 90 days to allow reanalysis should there by any question of the results.



DEFINITION

The analysis of purgeable organics consists of two parts: stripping and concentration of the organics by the purge and trap technique and subsequent analysis by GC/MS. Thirty volatile organics are determined over a concentration range of approximately 0.1 to 100 µg/L. Actual detection limits are dependent on the daily performance of the GC/MS system.

APPARATUS

Finnigan Series 4000 Gas Chromatograph Mass Spectrometer interfaced to Tekmar LSC-2 Sample Concentrator. The Finnigan 4021 GC/MS is a high performance low resolution quadruple mass spectrometer equipped with dynode conversion prior to the electron multiplier for high sensitivity. The Tekmar LSC-2 concentrator is an automated purge and trap device which is interfaced to the gas chromatograph of the GC/MS via a heated stainless steel transfer line.

REAGENTS:

- A. Organic-free water is generated by passing deionized distilled water through a stainless steel ultraviolet reaction vessel at the rate of 2 liters per half hour. The water is collected in a 2 L amber bottle and tightly sealed.
- B. Methanol, sodium thiosulfate and other reagents—ACS reagent grade.
- C. Standards: 95+ percent purity

STANDARD PREPARATION

A. Stock standard solutions

- 1. Fill a 50 ml, glass stoppered volumetric flask with approximately 49 ml of methanol. Let stand for a few minutes until weight is constant. Stopper the flask and weigh to ±0.1 mg.
- 2. Add 3-4 drops as required of the standard to the flask with a syringe or Pasteur pipette and reweigh. NOTE: The drops of the standard organic material should fall directly into the alcohol without contacting the flask neck.
- 3. Dilute to volume with methanol, stopper tightly and mix by inverting the flask 1-2 times.
- 4. Transfer aliquots of the solution into 2 ml ampules and seal. Transfer other aliquots to amber vials with teflon seals for immediate use. Calculate the concentration in \(\mu_0 / \mu \) from the net gain in weight.

B. Internal Standards

1. The internal standards 1,3-Dichloropropane and 1-Bromo-4-fluorobenzene are prepared in methanol by the same procedure.

SAMPLE CONCENTRATION AND ANALYSIS

- A. Spike the sample contained in the 60 ml amber bottle with the appropriate quantity of the internal standards in methanol to produce a concentration of 5 µg/L. Allow the internal standards to equilibrate within the sample for 1/2 hour prior to analysis.
- B. Rinse the 25 ml gas-tight syringe and the 25 ml sparger on the Tekmar concentrator with laboratory blank water.
- C. Pour the sample into the gas-tight syringe very gently and replace the plunger to bring the volume to exactly 25 ml.
- D. Load the sample into the Tekmar sparger via the luer lock valve without aerating the sample.
- E. Purge the sample for 8 minutes with a helium flow of 15 ml/min into the Tenax/silica gel trap which is maintained at 25°C.
- F. Set the mass spectrometer to run in the electron impact direct mode with an emission current of 0.5 milliamps, an electron energy of 70 electron volts, the electron multiplier at 1200 to 1800 volts (depending on the age of the electron multiplier), the dynodes at 3000 volts, and the ion source at 300°C. Set the data system to scan from m/e 34-300 in 0.75 seconds with a 0.05 second hold at the bottom.
- G. Apply liquid nitrogen in a styrofoam cup to the front of the capillary column in preparation of the upcoming desorption step.
- H. Desorb the compounds from the Tenax/silica gel trap at 200°C for 2.5 minutes with a 15 ml/min helium flow rate. The liquid nitrogen is removed following completion of the desorption step and the oven door closed.
- I. A split ratio of 10:1 is used during the desorption step.
- J. The gas chromatograph is programmed from the beginning of the desorption step. First the GC is held isothermal at 35°C for 4.5 minutes. Then the oven is programmed to 65° at 4°C/minute followed by 8°C/minute to 180°C where the temperature is held for the duration of the run.
- K. Between runs the Tenax trap is baked out at 225°C for 8 minutes.
- L. Injector interface and transfer line temperatures are maintained at 250°C throughout the concentration and analysis procedure.

CALCULATION

At the completion of the GC/MS run, the data are submitted to the Finnigan automated quantitation software analysis program. Identifications and quantification assigned by the automatic software is always checked manually by the mass spectroscopist for accuracy.

QUALITY ASSURANCE

Quality assurance is maintained by the following analytical routine:

- A. The GC/MS is tuned to meet 4-Bromofluorobenzene specifications as detailed in the Quality Control Manual.
- B. A laboratory water blank is analyzed to check for artifacts from the GC/MS system and/or for the presence of impurities in the water blank making it unsuitable for standard preparation.
- C. Field blanks are analyzed on a routine basis as a part of the quality assurance program.
- D. A standard containing all thirty purgeable organic compounds is analyzed and proper instrument sensitivity and stability are determined by comparison of performance with earlier calibration curves stored in the quantitation software. Significant deviation from previous quantitation curves requires running a complete new set of calibration standards.
- E. The precision of the technique is determined by running 10 percent of the samples in duplicate.
- F. The accuracy of the technique is determined by spiking 10 percent of the samples and determining recoveries.
- G. After manual review of the data generated by the Finnigan software, the mass spectroscopist submits a report to be typed by the lab clerk. The lab clerk proofs the report for accuracy after it is typed.
- H. The mass spectroscopist proofreads the report and signs it on the bottom when satisfied that the results reported are valid.
- I. All worksheets, chromatograms, quantitation reports, and calibration curves are organized and the final report submitted to the chief organic chemist for final review. As a final check, the values reported are compared to the typical concentration range expected for the particular water supply.
- J. The worksheets, chromatograms, quantitation reports, etc. are filed according to lab number by the lab clerk. The final report is duplicated and placed in the organics file report binder, also according to lab number.
- K. The final report is sent out.

L. The remaining portion of the water sample is stored for one month at 4°C to allow reanalysis should there be any question of the results.





DEFINITION

The analysis of base/neutrals and acids covers the determination of a number or organic compounds that are solvent-extractable and amenable to gas chromatography/mass spectrometry. Approximately 48 base/neutrals and 12 acid-extractables are determined over a concentration range of approximately 1-100 µg/L. Actual detection limits are dependent on the daily performance of the GC/MS system.

APPARATUS

STATES STATES CONCRETE SECTION

CONTRACT CONTRACT CONTRACT L'ACCION DIVINI VIN

Finnigan Series 4021 gas chromatograph/mass spectrometer. The project's Finnigan 4021 gas chromatograph/mass spectrometer is a low resolution, quadrapole mass spectrometer.

REAGENTS

- A. Organic-free water is generated by passing deionized distilled water through a stainless steel ultra-violet reaction chamber at 2 liters per half hour. This water is collected in a 2 liter amber bottle and tightly sealed.
- B. Methanol, sodium hydroxide, sulfuric acid, sodium sulfate and other reagents--ACS reagent grade.
- C. Standards ACS grade, 95+ percent purity where possible.

STANDARD PREPARATION

Stock standard solutions are prepared at a concentration of 1 µg/µl. These standards are prepared by dissolving the appropriate quantity of assayed reference material into a pesticide-quality solvent (appropriate) and diluting to volume in a 50 ml ground-glass stoppered volumetric flask. This primary stock solution is transferred to 2 ml amber ampules and/or amber glass teflon-sealed vials and stored at -20°C until used.

INTERNAL STANDARD

Internal standards are prepared using the same method as that for standards detailed above.

EXTRACTION

A. Base/neutrals

1. Pour sample into 1 liter graduated cylinder and transfer contents to a 2 liter separatory funnel.

- 2. Adjust the pH of the sample with 6 normal sodium hydroxide to pH 11 or greater. Use multi-range pH paper for the measurements, using a Pasteur pipette to transfer a drop of the sample to the paper.
- 3. Add 60 ml, pesticide-grade methylene chloride to the graduated cylinder to rinse the walls. Transfer the solvent into the separatory funnel and extract the sample by shaking the funnel for 2 minutes with periodic venting to release excess vapor pressure.
- 4. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample but may include stirring, filtration of emulsion through glass wool or centrifugation.
- 5. Add a second 60 ml volume of methylene chloride to the sample bottle and complete the extraction procedure a second time combining the extracts in an erlenmeyer flask.
- 6. Perform a third extraction in the same manner.
- 7. Pour the combined extract through a drying column containing 3-4 inches of anhydrous sodium sulfate and collect the eluant in a 500 ml Kuderna Danish (KD) flask equipped with a 10 ml concentrator tube.
- 8. Rinse the erlenmeyer flask with 20-40 ml of methylene chloride. Pour this through the drying column, adding to the total extract.
- 9. Add 1-2 clean Hengar boiling chips to the flask and attach a three-ball macro-Snyder column.
- 10. Prewet the Snyder column by adding about 1 ml methylene chloride through the top.
- 11. Place the KD apparatus on a warm water bath 60-65°C so that the concentrator tube is partially emersed in the water and the entire lower rounded surface of the flask is bathed with water vapor. Adjust the apparatus to a vertical position and the water temperature as required to complete the concentration in 20 minutes. NOTE: At the proper rate of distillation the balls of the column actively chatter but the chambers do not flood.
- 12. When the volume has reached an apparent volume of 1 ml, remove the KD apparatus and allow the solvent to drain for at least 10 minutes while cooling.
- 13. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1-2 ml of methylene chloride. A Pasteur pipette may be used for this operation.

- 14. Add a clean boiling chip and attach a two-ball micro-Snyder column to the concentrator tube.
- 15. Prewet the column by adding about 0.5 ml methylene chloride to the top.
- 16. Place the KD apparatus in a Kontes heating block and set the temperature so that the concentration is completed in 10 minutes.
- 17. When the liquid reaches an apparent volume of about 0.5 ml, remove the KD from the Kontes heater and allow the solvent to drain and cool for at least 10 minutes. Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with approximately 0.2 ml of methylene chloride.
- 18. Adjust the final volume to 1.0 ml, seal and label as the base/neutral fraction.

B. Acid fraction

STATE AND STATE STATES ASSESSED.

THE PROPERTY PROPERTY INCHES

- 1. Adjust the pH of the water previously extracted for base/neutrals with 6-normal sulfuric acid to pH 2 or below.
- 2. Serially extract with 60, 60 and 60 ml portions of pesticide grade methylene chloride.
- 3. Collect and combine the extracts in a 250 ml erlenmeyer flask and dry by passing through a column of anhydrous sodium sulfate.
- 4. Rinse the erlenmeyer flask with 20-40 ml of methylene chloride and pour through the drying column.
- 5. Concentrate the extract to a final volume of 1 ml, following the same procedure as outlined above for concentration of the base/neutral extract.

ANALYSIS

The base/neutral and acid fractions are analyzed with the following gas chromatograph and mass spectrometer conditions:

GC/MS SYSTEM

A. Gas chromatograph conditions

1. Column:

0.25 mm x 15 M fused silica open tubular (FSOT) column coated with DB-5 (J&W Scientific)

2. Column head pressure: 10 psi

3. Injector temperature: 250°C

4. Carrier gas: helium

5. Column temperature program: 1 minute at 65°C then 65-95°C at

20/minute followed by 95°C to 300°C at 80/minute. The temperature is held at 300°C for the remainder of

the run.

B. Mass spectrometer conditions

1. Separator oven: 250°C

2. Transfer line: 250°C

3. Mode: electron impact direct

4. Emission current: 0.5 milliamps

5. Electron energy: 70 eV

6. Electron multiplier: 1200-1800 Volts

7. Dynodes: 3000 Volts

8. Ion source temperature: 300°C

9. Data system scan: M/E 34-345 in 0.95 seconds with a

0.05 second hold at the bottom

C. A 10 µ syringe is thoroughly cleaned with methylene chloride.

D. 2 µl of the acid and/or base/neutral fraction is loaded into the syringe.

E. The sample is injected directly into the capillary column and the GC program and MS acquisition started.

CALCULATION

C. I. LONGER OFFICERS LINESPOSE LINESPOSE SOURCES STATEMENT SOURCES STATEMENT DESCRIPTION OF THE STATEMENT O

- A. At the end of the GC/MS run the data are submitted to analysis by the Finnigan reverse search software.
- B. Identifications and quantification values generated by the automated software are manually checked by the mass spectroscopist for validity.

QUALITY ASSURANCE

Quality assurance is maintained by the following analytical routine:

- A. The GC/MS is tuned to meet DFTPP specifications as detailed in the Quality Control Manual.
- B. A laboratory water blank is analyzed to check for artifacts from the GC/MS system for the presence of impurities in the water blank making it unsuitable for standard preparation.
- C. Field blanks are analyzed on a routine basis as a part of the quality assurance program.
- D. Standards containing all the extractable organic compounds are analyzed and proper instrument sensitivity and stability are determined by comparison of performance with earlier calibration curves stored in the quantitation software. Significant deviation from previous quantitation curves requires running a complete new set of calibration standards.
- E. The precision and accuracy of the technique is determined by running 10 percent of the samples either in duplicate, or with a standard spike addition.
- F. After manual review of the data generated by the Finnigan software, the mass spectroscopist submits a report to be typed by the lab clerk. The lab clerk proofs the report for accuracy after it is typed.
- G. The mass spectroscopist proofreads the report and signs it on the bottom when satisfied that the results reported are valid.
- H. The final report is submitted to the chief organic chemist for final review. As a final check, the values reported are compared to the typical concentration range expected for the particular water supply.
- I. The worksheets, chromatograms, quantitation reports, etc. are filed according to client by the lab clerk. The final report is duplicated and placed in the organics file report binder.
- J. The final report is sent out.

to property strooms incomes traction perform percent assessed appropriate appropriate and the second second

K. The remaining portion of the water sample is stored for one month at 4°C to allow re-analysis should there be any question of the results.

ANALYSIS OF HERBICIDES IN POTABLE AND NON-POTABLE WATER BY GC

DEFINITION

This analysis for herbicides includes 2,4-D, silvex, 2,4,5-T, and their corresponding acids, salts, and esters. These compounds are isolated from water by liquid-liquid extraction, derivitized and quantitated by gas chromatography in the concentration range from $0.1-10~\mu g/L$. Actual detection limits are dependent on instrumental stability and interferences present in the sample matrix.

APPARATUS

Varian 4600 GC equipped with capillary column and electron capture detector, controlled by a 401 data system.

REAGENTS

- A. Organic-free water is generated by passing deionized distilled water through a stainless steel ultra-violet reaction chamber at 2 liters per half hour. This water is collected in a 2 liter amber bottle and tightly sealed.
- B. Methylene chloride, toluene, acetone, and ethyl ether pesticide grade.
- C. Potassium hydroxide, sulfuric acid, sodium sulfate, and other reagents -ACS grade.
- D. Florisil P.R. grade.
- E. Standards EPA reference standards.

STANDARD PREPARATION

Stock standard solutions are prepared at a concentration of 1 µg/µl. These standards are prepared by dissolving the appropriate quantity of assayed reference material into a pesticide-quality solvent (appropriate) and diluting to volume in a ground-glass stoppered volumetric flask. This primary stock solution is transferred to 2 ml amber ampules and/or amber glass teflon-sealed vials and stored at -18°C until used.

INTERNAL STANDARD

Internal standards are not used for herbicide analysis.

EXTRACTION AND ESTERIFICATION

A. Pour 1 L of sample into a graduated cylinder and transfer the contents into a 2 L separatory funnel.



- B. Adjust the pH to 2 with concentrated sulfuric acid. Use multi-range pH paper for measurements, using a Pasteur pipette to transfer a drop of sample to the paper.
- C. Add 60 ml, pesticide-grade methylene chloride to the graduated cylinder and rinse the walls. Transfer the solvent into the separatory funnel and extract the sample by shaking the funnel for two minutes with periodic venting to release excess vapor pressure.
- D. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample but may include stirring, filtration of emulsion through glass wool or centrifugation.
- E. Add a second 60 ml volume of methylene chloride to the separatory funnel and complete the extraction procedure a second time combining the extracts in an erlenmeyer flask.
- F. Perform a third extraction in the same manner.

ELECTRIC PROPERTY DESCRIPTION DESCRIPTION

PARTICIPATE PARTICIPATE STREET, STREET, PARTICIPATE PROPERTY

- G. Pour the combined extract through a drying funnel containing 3-4 inches of acidified anhydrous sodium sulfate and collected in a 500 ml Kuderna Danish (KD) flask equipped with a 10 ml concentrator tube.
- H. Rinse the Erlenmeyer flask with 20-40 ml of methylene chloride. Pour this through the drying column, adding to the total extract.
- I. Add 15 ml 1N potassium hydroxide solution to the KD for hydrolysis.
- J. Add 1-2 clean Hengar boiling chips to the flask and attach a three-ball macro-Snyder column.
- K. Prewet the Snyder column by adding about 1 ml methylene chloride through the top.
- L. Place the KD apparatus on a warm water bath 80°C so that the concentrator tube is partially emersed in the water and the entire lower rounded surface of the flask is bathed with water vapor. Adjust the apparatus to a vertical position and heat for one hour.
- M. Remove the KD apparatus and allow the solvent to drain for at least 10 minutes while cooling.
- N. Remove the Snyder column and add 2 ml of 25 percent sulfuric acid.
- O. Transfer the contents of the KD flask to a 125 ml separatory funnel and extract three times with 20 ml volume of methylene chloride.

- P. Collect the combined methylene chloride extract in an Erlenmeyer flask. Pour the extracts through a funnel containing 5 grams acidified anhydrous sodium sulfate.
- Q. Transfer the dried methylene chloride extract to a KD apparatus and evaporate to approximatey 5 ml.
- R. Remove the KD from the water bath and allow to cool for 10 minutes. Remove the receiver from the KD apparatus and attach a micro-Snyder condenser.
- S. Add 1 ml of toluene and evaporate the extract to approximately 0.9 ml using a stream nitrogen and a hot water bath.
- T. Remove from heat and cool for 10 minutes. Add 0.5 ml fourteen percent boron trifluoride in methanol solution.
- U. Heat the micro KD apparatus in a Kontes heater block for 30-40 minutes at 55°C.
- V. Remove from heat and cool for 10 minutes. Add 4.5 ml five percent sodium sulfate solution, cap the receiver, and shake for 30 seconds and allow to stand for 10 minutes.
- W. Remove the toluene layer (top layer) and pass through a micro-column composed of a lower layer of 1.5 cm florisil and 2.0 cm acidifed sodium sulfate in a Pasteur pipette plugged with glass wool.
- X. Add 1 ml of toluene to the receiver containing the sodium sulfate solution. Repeat shaking, separation, and micro-column steps until a total of 5 ml of solvent have been collected.

ANALYSIS

THE PROPERTY PROBLEMS INTERESTED INTERESTED BEST OFFICERS OFFICERS BUTTON AND THE PARTY OF THE P

The general methodology outlined is taken from the 15th edition of <u>Standard Methods</u>. All samples are analyzed by capillary column.

GC SYSTEM

Chromatograph conditions

A. Column:

0.25 mm x 30 meter open tubular fused silica capillary column coated with DB-5 (J & W Scientific).

B. Column Temperatures:

Capillary column temperature program: 160°, 2 minutes hold, 6° per minute to 280°C.

C. Electron Capture Detector Temperature: 300°C

D. Injector Temperature: 220°C

E. Gases:

Carrier Gas ECD Make up Hydrogen (1 ml/min at 0.7 kg/cm²)

Nitrogen (30 ml/min)

F. Sample Injection: Samples are injected using a Model 8000 Varian autosampler. The injections are 2 µl. The syringes are cleaned thoroughly with pesticide grade hexane prior to each injection.

CALCULATION

このなる かいかいかい かんかんちゅう カンドカンスカー でんかんしゅう マンドアファン

CENTRAL CONTROL INVOVEMENT PROBLEM CONTROL

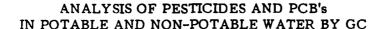
Calculations are made using a Varian Vista 401 data station which compares retention times and peak areas to those of an analytical standard run in the same manner as the sample extracts.

QUALITY ASSURANCE

- A. With each set of samples, a laboratory water sample is spiked with a standard mixture, carried through the extraction procedure, and analyzed by GC. Recoveries of herbicides are compared to previously developed precision/accuracy and recovery data. Significant deviation from previous quantitation data requires running a complete new set of standards.
- B. A laboratory water blank is extracted and analyzed with each set of samples processed to ensure the absence of interfering artifacts.
- C. Field blanks are analyzed on a routine basis as a part of the quality assurance program.
- D. The precision and accuracy of the technique is determined by running 10 percent of the samples either in duplicate, or with a standard spike addition.
- E. After manual review of the data generated by the GC software, the analyst submits a report to be typed by the lab clerk. The lab clerk proofs the report for accuracy after it is typed.
- F. The analyst proofreads the report and signs it on the bottom when satisfied that the results reported are valid.
- G. The final report is submitted to the chief organic chemist for final review. As a final check, the values reported are compared to the typical concentration range expected for the particular water supply.
- H. The worksheets, chromatograms, quantitation reports, etc. are filed according to client by the lab clerk. The final report is duplicated and placed in the organics file report binder, also according to client.

I. The final report is sent out.

J. The remaining portion of the water sample is stored for one month at 4°C to allow reanalysis should there be any question of the results.



DEFINITION

The analysis of pesticides and PCB's covers approximately 23 compounds that are solvent-extractable and determined over a concentration range of 0.01 to 10 µg/L. Actual detection limits are dependent on instrument stability and interferences in the sample.

APPARATUS

Varian 4600 GC with capillary column electron capture and nitrogen phosphorus detectors, controlled by a Varian 401 Data System.

REAGENTS

- A. Organic-free water is generated by passing deionized distilled water through a stainless steel ultraviolet reaction chamber at 2 liters per half hour. This water is collected in a 2 liter amber bottle and tightly sealed.
- B. Methylene chloride, hexane, methanol, acetone, and ethyl ether pesticide grade.
- C. Sodium hydroxide, sulfuric acid, sodium sulfate, and other reagents -ACS grade.
- D. Florisil P.R. grade.
- E. Standards EPA reference standards.

STANDARD PREPARATION

Stock standard solutions are prepared at a concentration of 1 µg/µl. These standards are prepared by dissolving the appropriate quantity of assayed reference material into a pesticide-quality solvent (appropriate) and diluting to volume in a ground-glass stoppered volumetric flask. This primary stock solution is transferred to 2 ml amber ampules and/or amber glass teflon-sealed vials and stored at -18°C until used.

INTERNAL STANDARD

Internal standards are not used for pesticide analysis because of potential interference from the multicomponent compounds (toxaphene, chlordane, PCB's).

TRACECCOCCE LANGUAGE

EXTRACTION

A. Pour 1 liter of sample into a graduated cylinder and transfer the contents into a 2 liter separatory funnel.



ACCOUNT ASSESSED TO SUSPEND INCOME AND ACCOUNT

- B. Add 60 ml, pesticide-grade methylene chloride to the graduated cylinder and rinse the walls. Transfer the solvent into the separatory funnel and extract the sample by shaking the funnel for 2 minutes with periodic venting to release excess vapor pressure.
- C. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample but may include stirring, sonification, or filtration of emulsion through glass wool or centrifugation.
- D. Add a second 60 ml volume of methylene chloride to the separatory funnel and complete the extraction procedure a second time combining the extracts in an erlenmeyer flask.
- E. Perform a third extraction in the same manner.

THE PARTY OF THE PROPERTY STATES OF STATES

- F. Pour the combined extract through a drying column containing 3-4 inches of anhydrous sodium sulfate and collected in a 500 ml Kuderna Danish (KD) flask equipped with a 10 ml concentrator tube.
- G. Rinse the erlenmeyer flask with 20-40 ml of methylene chloride. Pour this through the drying column, adding to the total extract.
- H. Add 1-2 clean Hengar boiling chips to the flask and attach a three-ball macro-Snyder column.
- I. Prewet the Snyder column by adding about 1 ml methylene chloride through the top.
- J. Place the KD apparatus on a warm water bath at 60-65°C so that the concentrator tube is partially emersed in the water and the entire lower rounded surface of the flask is bathed with water vapor. Adjust the apparatus to a vertical position and the water temperature as required to complete the concentration in 20 minutes. NOTE: At the proper rate of distillation the balls of the column actively chatter but the chambers do not flood.
- K. When the volume has reached an apparent volume of 1 ml, remove the KD apparatus and allow the solvent to drain for at least 10 minutes while cooling.
- L. Remove the Snyder column and add 50 ml hexane to the flask.
- M. Add a clean boiling chip and reattach the Snyder column.
- N. Prewet the column by adding about 0.5 ml hexane to the top.
- O. Place the KD apparatus in a warm water bath and set the temperature to 90°C so that the concentration is completed in 10 minutes.



LA LES LES LIGHTON DE LA LICHTE BANKS AND LA LICHTE BANKS AND LA LICHTE BANKS AND LA LICHTER BANKS AND LA LICHTER

- P. When the liquid reaches an apparent volume of about 2 ml, remove the KD from the water bath and allow the solvent to drain and cool for at least 10 minutes. Remove the Snyder column and rinse its lower joint into the concentrator tube with approximately 2 ml of hexane.
- Q. Adjust the final volume to 5ml by blowing under nitrogen. The extract is then transferred to a 5 ml glass vial with TFE septum.

ANALYSIS

Pesticide analysis is performed using EPA Method 608 as the basic guideline. All samples are analyzed using capillary columns. If interferences are present that impact identification and quantification then samples are cleaned up using Florisil (see EPA Method 608). Confirmation is performed using a second capillary column.

GC SYSTEM

- A. Gas chromatograph conditions
 - 1. Columns:
 - a) 0.25 mm x 30 meter open tubular fused silica capillary column coated with SE-54 (J & W Scientific).
 - b) 0.25 m x 30 meter open tubular fused silica capillary column coated with DB-1701 (J & W Scientific) for confirmation.
 - 2. Column Temperature:

Temperature program 55°C for 1 minute then programmed to 155°C at 25°C/min. The column is then programmed to 250°C at 6°C/min and held there for 1 to 5 minutes.

- 3. Detector Temperature: 300°C
- 4. Injector Temperatures: 220°C
- 5. Gases:
 - a) Carrier Gas: Hydrogen (1 ml/min at 0.7 kg/cm²)
 - b) ECD make up: Nitrogen (30 ml/min)
- 6. Sample Injection: Samples are injected onto the column using a Model 8000 Varian autosampler. The injection volume is 2 µl splitless. The syringes are cleaned thoroughly with pesticide grade hexane prior to each injection.

CALCULATION

CHANGE PREPARE ANDRESS CHARACT

CANADA CONTRACTOR OF THE PROPERTY OF THE PROPE

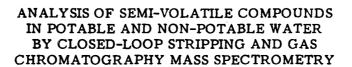
Calculations are made using a Varian Vista 401 data station which compares retention times and peak areas to those of an analytical standard run in the same manner as the sample extracts.

QUALITY ASSURANCE

- A. With each set of samples, a laboratory water sample is spiked with a standard mixture, carried through the extraction procedure, and analyzed by GC. Recoveries of pesticides/PCB's are compared to previously developed precision/accuracy and recovery data. If significant deviation from previous quantitation data occurs, the average of the previous recoveries is used.
- B. A laboratory water blank is extracted and analyzed with each set of samples processed to ensure the absence of interfering artifacts.
- C. The precision and accuracy of the technique is determined by running 10 percent of the samples either in duplicate, or with a standard spike addition.
- D. After manual review of the data generated by the GC software, the analyst submits a report to be typed by the lab clerk. The lab clerk proofs the report for accuracy after it is typed.
- E. The analyst proofreads the report and signs it on the bottom when satisfied that the results reported are valid.
- F. The final report is submitted to the senior organic chemist for final review.

 As a final check, the values reported are compared to the typical concentration range expected for the particular water supply.
- G. The worksheets, chromatograms, quantitation reports, etc. are filed according to client by the analyst. The final report is duplicated and placed in the organics files, also according to client.
- H. The final report is sent out.
- I. The remaining portion of the water sample is stored for one month at 4°C to allow reanalysis should there be any question of the results.





DEFINITION

CONTRACTOR CONTRACTOR SAMPLES CONTRACTOR CON

ACTION TO THE PARTY OF THE PART

The analysis of semi-volatile organics consists of two parts: stripping and concentration of the organics by the closed-loop stripping technique and subsequent analysis by GC/MS. Over 50 semi-volatile organics are determined over a concentration range of approximately 0.1 to 1000 ng/L. Actual detection limits are dependent on the daily performance of the closed-loop stripping apparatus and the GC/MS system.

APPARATUS

A closed-loop stripping apparatus fashioned after that of Grob (J. Chromotography 84:255 (1973)) was built in the lab workshop. The extracts from this device are injected into a Finnigan 4021 GC/MS.

REAGENTS

- A. Organic-free water is generated by passing deionized distilled water through a stainless steel ultraviolet reaction vessel at the rate of 2 liters per half hour. The water is collected in a 2 liter amber bottle and tightly sealed.
- B. Solvents: Acetone JT Baker Resi-Analyzed; Methylene Chloride JT Baker Resi-Analyzed; Carbon Disulfide Aldrich Gold Label 99+ percent.
- C. Sodium sulfate, sodium thiosulfate and other reagents ACS reagent grade.
- D. Standards: 95+ percent purity

STANDARD PREPARATION

A. Stock standard solutions

- 1. Fill a 50 ml, glass stoppered volumetric flask with approximately 49 ml of methylene chloride. Let stand for a few minutes until weight is constant. Stopper the flask and weigh to +0.1 mg.
- 2. Add 3-4 drops as required of the standard to the flask with a syringe or Pasteur pipette and reweigh. Note: The drops of the standard organic material should fall directly into the solvent without contacting the flask neck.
- 3. Dilute to volume with methylene chloride, stopper tightly and mix by inverting the flask one to two times.

4. Transfer aliquots of the solution into 2 ml ampules and seal. Transfer other aliquots to amber vials with teflon seals for immediate use. Calculate the concentration in mg/ml from the net gain in weight.

B. Internal Standards

1. The internal standards, 1-bromohexane and 1-chlorodecane, are prepared in methylene chloride by the same procedure.

SAMPLE CONCENTRATION

- A. 500 ml of the sample is poured into a clean 500 ml volumetric flask. The sample is spiked with internal standard to produce a concentration of 100 ng/L.
- B. The sample is poured into the CLS vessel and 20 gm sodium sulfate is added.
- C. A freshly cleaned carbon trap is inserted into its holder.
- D. The system is closed and the sample purged for one hour at 60°C with stirring.
- E. The carbon trap is removed and extracted twice with 10 carbon disulfide. Sixteen it are recovered and stoe to two times.
- F. The CLS vessel is thoroughly rinsed with clean water (and methanol if required) before the next run.

ANALYSIS

THE PROPERTY OF THE PROPERTY O

The CLS extracts are analyzed with the following gas chromatograph and mass spectrometer conditions:

GC/MS SYSTEM

A. Gas chromotograph conditions

1. Column:

0.25 mm x 15 M fused silica open tubular (FSOT) column coated with DB-5 (J & W Scientific)

2. Column head pressure: 8 psi

3. Injector temperature: 250°C

4. Carrier gas: helium

5. Column temperature program: Column oven door open for one minute then hold temperature at

50°C for one minute and program from 50°C to 95°C to 280°C at

60/minute. The temperature is held at 280°C for the remainder of the run

B. Mass spectrometer conditions

1. Separator oven: 290°C

2. Transfer line: 290°C

3. Mode: electron impact direct

4. Emission current: 0.5 milliamps

5. Electron energy: 70 eV

6. Electron multiplier: 1200-1800 volts

7. Dynodes: 3000 volts

8. Ion source temperature: 250°C

9. Data system scan: M/E 34-450 in 0.95 seconds with a

0.05 second hold at the bottom

C. 2 µl of extract are injected into the column and 3700 scans acquired.

QUALITY ASSURANCE

THE PARTY CONTROL OF THE PROPERTY WHENCE STATES BRINGS TOWN

Quality assurance is maintained by the following analytical routine:

- A. The GC/MS is tuned to meet 4-Bromofluorobenzene specifications as detailed in the Quality Control Manual.
- B. A laboratory water blank is analyzed to check for artifacts from the GC/MS system and/or for the presence of impurities in the water blank making it unsuitable for standard preparation.
- C. Field banks are analyzed on a routine basis as a part of the quality assurance program.
- D. A standard containing all 50 semi-volatile organic compounds is analyzed and proper instrument sensitivity and stability are determined by comparison of performance with earlier calibration curves stored in the quantitation software. Significant deviation from previous quantitation curves requires running a complete new set of calibration standards.
- E. The precision and accuracy of the technique is determined by running 10 percent of the samples in duplicate or spiked with standards.

- F. After manual review of the data generated by the Finnigan software, the mass spectroscopist submits a report to be typed by the lab clerk. The lab clerk proofs the report for accuracy after it is typed.
- G. The mass spectroscopist proofreads the report and signs it on the bottom when satisfied that the results reported are valid.
- H. The final report is submitted to the chief organic chemist for final review.

 As a final check, the values reported are compared to the typical concentration range expected for the particular water supply.
- I. The worksheets, chromatograms, quantitation reports, etc. are filed according to client by the lab clerk. The final report is duplicated and placed in the organics file report binder, also according to client.
- J. The final report is sent out.

THE PARTY ASSESSED THE STATE OF THE STATE OF THE STATE STATE STATE STATE STATE STATES

K. The remaining portion of the water sample is stored for one month at 4°C to allow reanalysis should there be any question of the results.





DEFINITION

CHARLES HOUSE

Secretary Agreement Secretary Secretary

The analysis of Volatile Polar Organics covers the determination of a number of organic compounds that can be steam distilled and are amenable to gas chromatography/mass spectrometry. Numerous compounds are determined over a concentration range of approximately 0.1 to 100 µg/L. Precision and accuracy data has been developed for eight specific compounds. Detection limits are dependent on the daily performance of the GC/MS system.

APPARATUS

Finnigan Series 4000 Gas Chromatograph Mass Spectrometer interfaced to Tekmar LSC-2 Sample Concentrator. The Finnigan 4021 GC/MS is a high performance low resolution quadrupole mass spectrometer equipped with dynode conversion prior to the electron multiplier for high sensitivity. The Tekmar LSC-2 concentrator is an automated purge and trap device which is interfaced to the gas chromatograph of the GC/MS via a heated stainless steel transfer line. The distillation equipment consist of a two liter round bottom flask, 300 mm Vigreaux distallation column, West condenser, vacuum adapter and 15 ml pear shaped receiver.

REAGENTS

- A. Organic-free water is generated by assing deionized distilled water through a stainless steel ultra-violet reaction chamber at 2 liters per half hour. This water is collected in a 2 liter amber bottle and tightly sealed.
- B. Sodium Sulfate ACS reagent grade.
- C. Standards ACS grade, 95+ percent purity where possible.

STANDARD PREPARATION

Stock standard solutions are prepared at a concentration of 1 µg/µl. These standards are prepared by dissolving the appropriate quantity of assayed reference material into purified deionized water and diluting to volume in a 50 ml ground-glass stoppered volumetric flask. This primary stock solution is transferred to 2 ml amber ampules and/or amber glass teflono-sealed vials and stored at -20°C until use.

DISTILLATION

- A. Pour sample into a one liter graduated cylinder and transfer the contents to a two liter round bottom flask.
- B. Add 40 grams sodium sulfate to the sample flask.

- C. Slowly begin distilling the sample. Distillation should take approximately one hour.
- D. Collect distillate in the 10 ml receiving flask which has been cooled in an ice bath to reduce the loss of the volatile compounds.
- E. Stop distillation when 4 mls have been collected. Allow apparatus to cool for 5 minutes. Final volume should be between 5 10 mls.
- F. Transfer distillate to 10 ml amber glass vial with TFE seal. Store at 4°C until analysis.

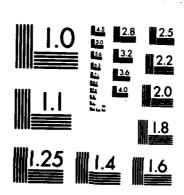
ANALYSIS

THE PARTY OF THE P

- A. One gram of sodium sulfate is added to the sample in the 10 ml amber glass vial.
- B. Rinse the 5 ml gas-tight syringe and the 5 ml sparger on the Tekmar concentrator with laboratory blank water.
- C. After the sodium sulfate has dissolved, pour the sample into the gas-tight syringe very gently and replace the plunger to bring the volume to exactly 5 ml.
- D. Inject into Tekmar LSC-2 and analyze as a VOA sample.



AD-8136 865	OPERATION POTOMAC N ENGINEER:	ESTUARY	' E (I	U) MON	TGOMER	RY (JAI	MES M)	CONSU	LTING	2/	6
UNCLÄSSIFIED	MMH-83-M	-VOL-1	DACH	31-80-	C-0041		il iki	F/G 1	3/2	NL	
7											



THE PROPERTY OF THE PROPERTY O

MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963-A



ACCOUNT OF THE PARTY OF THE PAR

ANALYSIS OF ORGANIC ANIONS IN POTABLE AND NON-POTABLE WATER BY ION EXCHANGE AND GC/MS

DEFINITION

The analysis of organic anions in water covers the determination of five compounds which can be adsorbed to an ion exchange resin and detectable by gas chromatography/mass spectrometry (GC/MS). The compounds are detectable over a concentration range of approximately 50 - 100 µg/L. The actual detection limits are dependent on the daily performance of the GC/MS system.

APPARATUS

The project's Finnigan 402l gas chromatograph mass spectrometer is a high performance, low resolution, quadrapole mass spectrometer equipped with dynode conversion for high sensitivity.

REAGENTS

- A. AG1-X8 (50-100 mesh) ion exchange resin (Biorad Laboratories, Richmond, CA) in chloride form.
- B. Distilled-deionized water
- C. Geseous HCl in lecture bottle Electronics grade
- D. Methanol, diethyl ether, ethanol (J.T. Baker Pesticide grade)
- E. Sodium Hydroxide, Potassium Hydroxide, phenolphthalein (A.C.S. reagent grade)
- F. Diazold (N-methyl-N-nitroso-p-toluenesulfonamide) (Aldrich Chemical Company)
- G. Standards ACS Grade, 95 percent + purity

STANDARD PREPARATION

Stock standard solutions are prepared at a concentration of l µg/ml. These standards are prepared by dissolving the appropriate quantity of assayed reference material into a pesticide-quality solvent (appropriate), and diluting to volume in a 50 ml ground-glass stoppered volumetric flask. This primary stock solution is transferred to 2 ml amber ampules and/or amber glass teflon-sealed vials and stored at -20°C until used.

SAMPLE PREPARATION

A. Preparation of the HCl:Methanol solution: The HCl:methanol solution is prepared in large batches and stored in the refrigerator up to 2 weeks before use. Gaseous HCl is bubbled through the methanol for approximately 15



minutes. The HCl concentration of the methanol solution is determined by adding 1 ml of the methanol solution to 20 ml of water, and titrating to the phenolphthalein endpoint with 1N KOH. A normality from 0.9 to 1.1 is acceptable for the analytical operation.

- B. Preparation of ethereal alcoholic solutions of diazomethane: Ethanol (25 ml) is added to a solution of potassium hydroxide (5 g) in water (8 ml) in a 100-ml distilling flask fitted with dropping funnel and an efficient condenser set downward for distillation. The condenser is connected to a cooled (°C) receiving flak. The flask containing the alkali solution is heated in water bath to 65°C, and a solution of Diazald (21.5 g) in about 200 ml of ether is added through the dropping funnel in about 25 min. When the dropping funnel is empty, another 40 ml of ether is added slowly and the distillation is continued until the distilling ether is colorless. The ethereal distillate contains about 3 g of diazomethane.
- C. Preparation of resin material: Biorad AG1-X8 resin is extracted with methanol in a Soxhlet apparatus overnight. The resin is placed in a column and rinsed with 10 bed volumes of water, 20 bed volume of lN NaOH, and 2 bed volumes of 1N formic acid. The resin is then rinsed with water until the eluent water is at pH 5 to 6. Resin which is not used immediately may be stored wet sealed in glass jars.

D. Sample Concentration:

- 1. Glass chromatograph columns, 10.5 mm i.d. x 25 cm, equipped with 250 ml reservoir and teflon stopcock were used.
- 2. Prepare ion-exchange column by placing a small glass wool plug in the bottom of the chromatography column. Pipette 10 ml (measure volume after resin has settled) of cleaned Biorad AG1-X8 resin into the columns, allow the resin to settle, and rinse the column with approximately 10 ml of water.
- 3. Prior to analysis, samples should stand undisturbed for 4 hours or overnight to allow particulates to settle.
- 4. One liter of water sample is allowed to drain through the resin bed. Using the stopcock at the base of the column, adjust flow to 2.5 ml/min.
- 5. Rinse the resin with 25 ml water. Allow the water to drain out of the resin bed, aspirate off remaining water from the bottom of the resin column.
- 6. Elute acids from the column using 100 ml of 1N HCl:methanol. Collect the eluate in a 200 ml round bottom flask. Elute at approximately 5 ml/min.
- 7. Remove all of the eluent using rotary evaporation at 30°C. Resuspend the sample residue in 3 ml of methanol and transfer to a 15 ml test

tube. Rinse the flask with two 2 ml portions of methanol and transfer to the test tube.

- 8. Evaporate the solvent to dryness using nitrogen blowdown at 40°C.
- 9. Resuspend the sample in 0.2 ml of lN HCl:methanol solution. Sample may be stored at this point.

E. Derivatization:

- 1. Add the diazomethane solution to the sample using a 1 ml disposable pipette. Continue adding solution until a yellow color persists. Excess diazomethane is evaporated off using nitrogen blowdown. If excess diazomethane has been prepared it should be quenched with a formic acid solution before discarding.
- 2. Concentrate the derivatized sample to 1 ml in the test tube using nitrogen blowdown at 40°C in a well-ventilated hood.

GC/MS SYSTEM

A. Gas chromatograph conditions

1. Column: 0.25 mm x 15 M fused silica open

tubular (FSOT) column coated with

DB-5 (J&W Scientific).

2. Column head pressure: 10 psi

3. On-column injector temperature: 250°C

4. Carrier gas: helium

5. Column temperature program: 1 minute at 80°C then program to

275°C at 10°C/min. The temperature is held at 275°C to the end of

the run.

B. Mass spectrometer conditions

1. Separator oven: 250°C

2. Transfer line: 250°C

3. Mode: electron impact direct

4. Emission current: 0.5 milliamps

5. Electron energy: 70 eV

6. Electron multiplier: 1200 - 1800 volts

7. Dynodes: 3000 Volts

8. Ion source temperature: 300°C

9. Data system scan: M/E 34-450 in 0.95 seconds with a

0.05 second hold at the bottom.

- C. A 10 µl sample syringe is thoroughly cleaned with methylene chloride.
- D. Two µ of the anionic methylated extract are loaded into the syringe.
- E. The sample is injected into the GC/MS and 2000 scans are acquired.

CALCULATION

STATES (CONTROL OF STATES STATES STATES STATES AND THE STATES STATES STATES STATES STATES STATES STATES

- A. At the end of the GC/MS run the data are submitted to analysis by the Finnigan reverse search software.
- B. Identifications and quantification values generated by the automated software are manually checked by the mass spectroscopist for validity.

QUALITY ASSURANCE

Quality assurance is maintained by the following analytical routine:

- A. The GC/MS is tuned to meet DFTPP specifications as detailed in the Quality Control Manual.
- B. A laboratory water blank is analyzed to check for artifacts from the GC/MS system for the presence of impurities in the water blank making it unsuitable for standard preparation.
- C. Field blanks are analyzed on a routine basis as a part of the quality assurance program.
- D. Standards containing all the extractable organic compounds are analyzed and proper instrument sensitivity and stability are determined by comparison of performance with earlier calibration curves. Significant deviation from previous quantitation curves requires running a complete new set of calibration standards.
- E. The precision and accuracy of the technique is determined by running 10 percent of the samples either in duplicate, or with a standard spike addition.
- F. After manual review of the data generated by the Finnigan software, the mass spectroscopist submits a report to be typed by the lab clerk. The lab clerk proofs the report for accuracy after it is typed.
- G. The mass spectroscopist proofreads the report and signs it on the bottom when satisfied that the results reported are valid.



- H. The final report is submitted to the senior organic chemist for final review.

 As a final check, the values reported are compared to the typical concentration range expected for the particular water supply.
- I. The worksheets, chromatograms, quantitation reports, etc., are filed according to client by the analyst. The final report is duplicated and placed in the organics files, also according to client.
- J. The final report is sent out.
- K. The remaining portion of the water sample is stored for one month at 4°C to allow re-analysis should there be any question of the results.

ANALYSIS OF ORGANIC CATIONS IN POTABLE AND NON-POTABLE WATER BY ION EXCHANGE AND HPLC

DEFINITION

The analysis of organic cations in water covers the determination of eight compounds which can be isolated from water on an ion exchange resin and detected by High Performance Liquid Chromatography (HPLC). The compounds are detectable over a concentration range of approximately 1 to 1000 µg/L. The actual detection limits are dependent on the daily performance of the HPLC system.

APPARATUS

WEST STREET, STREET, STREET, STREET, STREET,

THE PROPERTY SHOWING MAKERIANS I SHOWING THE PROPERTY

A Varian Model 54 High Performance Liquid Chromatograph is utilized for this analysis. The HPLC is equipped with two optical (UV/VIS) and one fluorescence spectrophotometric detectors. A reversed-phase MicroPak MCH-5 chromatographic column is employed to separate the components in the sample.

REAGENTS

- A. Sepralyte C-18 (40m) bonded phase silica-based resin (Analytichem International, Harbor, CA).
- B. Distilled/deionized water.
- C. Methanol (J.T. Baker Pesticide grade).
- D. Solvents for HPLC analysis: acetonitrile, methanol, and water (J.T. Baker HPLC grade).
- E. Potassium phosphate monobasic, phosphoric acid, sodium 1-heptanesulfonate (ACS reagent grade).
- F. Standard chemicals, 95+ percent purity.

STANDARD PREPARATION

A stock standard solutions (2 µg/ml) of each of the following organic cations is prepared in methanol (pesticide grade) solvent:

Benzidine
Pyridine
Indole
3-Chloropyridine

Quinoline

5-Chloroindole
3.3'-Dichlorobenzidine

4-Chloroquinoline



These standards are prepared by dissolving the appropriate quantity of assayed reference material into methanol and diluting to volume in a 50 ml ground-glass stoppered volumetric flask. This primary stock solution is transferred to 2 ml amber ampules and/or amber glass teflon-sealed vials and stored at -20°C until used.

TESSECTES SANCE DESCRIPTION

Programme Consideration (Consideration Consideration)

SAMPLE PREPARATION

- A. Glass chromatograph column, 10.5 mm i.d. x 25 cm, equipped with 250 ml reservoir and teflon stopcock were used.
- B. Prepare chromatograph column by placing a small glass wool plug in the bottom of the column. Fill the column with pesticide grade methanol, then add 0.75 g of Sepralyte C-18 resin into the column. Allow the resin to settle.
- C. Rinse the resin with 50 ml distilled/deionized water, followed with 50 ml of pesticide grade methanol, and finally with 50 ml of distilled/deionized water.
- D. Prior to analysis, samples should stand undisturbed for 4 hours or overnight to allow particulates to settle.
- E. One liter of water sample is allowed to drain through the resin bed. Using the stopcock at the base of the column, adjust flow to 2.5 ml/min.
- F. Rinse the resin with 25 ml water. Allow the water to drain out of the resin bed.
- G. Elute organic cations from the column using 100 ml of pesticide grade methanol. Collect the eluate in a 200 ml round bottom flask. Elute at approximately 5 ml/min.
- H. Concentrate the eluant solvent to 1 ml using rotary evaporation at 40°C. Sample may be stored at this point.

HPLC SYSTEM

A. Mobile phase:

- 1. Mode = isocratic
- 2. Flow rate = 0.6 ml/min.



3. Mobile phase composition:

35 percent-ion-pairing buffer solution (pH=3): 0.005 M sodium l-heptanesulfonate/0.05 M potassium phosphate monobasic/phosphoric acid.

55 percent - Methanol

10 percent - Acetonitrile

- B. Column: Varian MicroPak MCH-5 5 nm, 0.26 x 15 cm, HC-ODS Column (at 35°C).
- C. UV detector: max = 254 nm
- D. Fluorescence detector: ex 220 nm, em = 530 nm
- E. Sample volume: 10 µl of the cationic concentrated eluant is injected for HPLC analysis.

CALCULATION

- A. At the end of the HPLC run, the data are submitted to analysis by the Varian 401 data processor.
- B. Identifications and quantification values generated by the automated software are manually checked by the HPLC spectroscopist for validity.

QUALITY ASSURANCE

Quality assurance is maintained by the following analytical routine:

- A. A laboratory water blank is analyzed to check for artifacts from the HPLC system and/or for the presence of impurities in the water blank making it unsuitable for standard preparation.
- B. The precision of the technique is determined by running 10 percent of the samples in duplicate.
- C. The accuracy of the technique is determined by spiking 10 percent of the samples and determining recoveries.
- D. The HPLC analyst proofreads the report and signs it on the bottom when satisfied that the results reported are valid.
- E. All worksheets, chromatograms, quantitation reports, and calibration curves are organized and the final report and submitted to the senior organic chemist for final review. As a final check, the values reported are compared to the typical concentration range expected for the particular water supply.

- F. The worksheets, chromatograms, quantitation reports, etc., are fixed according to client by the analyst. The final report is duplicated and placed in the organics files.
- G. The final report is sent out.
- H. The remaining portion of the water sample is stored for one month at 4°C to allow reanalysis should there be any question of the results.

ANALYSIS OF POLYNUCLEAR AROMATIC HYDROCARBONS IN WATER BY HIGH PRESSURE LIQUID CHROMATOGRAPHY

DEFINITION

The analysis of polynuclear aromatic compounds (PNA) in water is conducted in three stages: (1) extraction of the organic compounds from aqueous solution with methylene chloride; (2) liquid chromatographic sample cleanup using silica gel for fractionation of the PNA derivatives from aliphatic and phenolic species; and (3) analysis of the sample extract by high performance liquid chromatography (HPLC).

APPARATUS

The instrument utilized for the analysis of PNA species is the Varian Model 54 High Performance Liquid Chromatograph. The HPLC is equipped with two absorbance ultraviolet (UV) and visible (VIS) detectors and one fluorescence spectrophotometric detector. A reversed-phase C18 chromatographic column is employed to separate the various polynuclear aromatic hydrocarbons for analysis.

REAGENTS

- A. Solvents for liquid-liquid extraction (LLE) and LC clean-up preparation of standards: acetonitrile, methylene chloride, pentane (pesticide grade).
- B. Solvents for HPLC Analysis: acetonitrile, and water (J.T. Baker HPLC grade).
- C. Standard chemicals: 95+ percent purity
- D. Sodium hydroxide, sulfuric acid (ACS reagent grade).

STANDARD PREPARATION

A. A stock solution (20 ppm) of each of the following PAH's is prepared in acetonitrile:

Naphthalene Benzo(g,h,i)perylene Fluorene Caffeine 2-Chloronaphthalene Phenanthrene Coronene Chrysene Anthracene Benzo(b)fluoranthene Pyrene Benzo(k)fluoranthene Benzo(a)pyrene Indo(1,2,3-c,d)pyrene Benzo(a)anthracene Dibenz(a,h)anthracene Fluoranthene 1,2,3,4-Dibenzanthracene

A sample of each reagent is weighed to ± 0.1 mg and placed in an amber glass 100 ml volumetric flask. Sufficient acetonitrile is added in order to ensure complete dissolution of each compound. The solution is then diluted

- to volume with acetonitrile. The flask is stoppered for storage at 4°C in the dark. Under these conditions, the solution should remain stable for 90 days.
- B. A dilute standard is prepared for calibration of the HPLC using the concentrated stock solution. The final concentration is 10 ng/10 ml in acetonitrile.

EXTRACTION

Market Straight Applied Children

markets the second of the seco

- A. Pour 1 L of sample into a graduated cylinder and transfer the contents into a 2 L separatory funnel.
- B. Adjust the pH to 7 using concentrated sulfuric acid or sodium hydroxidencessary. Use multi-range pH paper for measurements, using a Pas pipette to transfer a drop of sample to the paper.
- C. Add 60 ml, pesticide-grade methylene chloride to the graudated cylinder rinse the walls. Transfer the solvent into the separatory funnel and extract the sample by shaking the funnel for two minutes with periodic venting to release excess vapor pressure.
- D. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample but may include stirring, filtration of emulsion through glass wool or centrifugation.
- E. Add a second 60 ml volume of methylene chloride to the separatory funnel and complete the extraction procedure a second time combining the extracts in an erlenmeyer flask.
- F. Perform a third extraction in the same manner.
- G. The extracts are combined in a 250 ml round bottom flask. 1 gram of anhydrous magnesium sulfate is added.
- H. After 15 minutes the sample is filtered through a Whatman qualitative ashfree filter into a 500 ml Kuderna Danish (KD) flask equipped with a 10 ml concentrator tube.
- I. The residual MgSO₄ is washed with an additional 30 ml of methylene chloride. This is also drained into the KD.
- J. Add 1-2 clean Hengar boiling chips to the flask and attach a three-ball macro-Snyder column.
- K. Prewet the Snyder column by adding about 1 ml methylene chloride through the top.

- L. Place the KD apparatus on a warm water bath 60-65°C so that the concentrator tube is partially emersed in the water and the entire lower rounded surface of the flask is bathed with water vapor. Adjust the apparatus to a vertical position and the water temperature as required to complete the concentration in 20 minutes. NOTE: At the proper rate of distillation the balls of the column actively chatter but the chambers do not flood.
- M. When the volume has reached an apparent volume of 1 ml, remove the KD apparatus and allow the solvent to drain for at least 10 minutes while cooling.
- N. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1-2 ml of methylene chloride. A Pasteur pipette may be used for this operation.
- O. Add a clean boiling chip and attach a two-ball micro-Snyder column to the concentrator tube.
- P. Prewet the column by adding about 0.5 ml methylene chloride to the top.
- Q. Place the KD apparatus in a Kontes heating block and set the temperature so that the concentration is completed in 10 minutes.
- R. When the liquid reaches an apparent volume of about 0.5 ml, remove the KD from the Kontes heater and allow the solvent to drain and cool for at least 10 minutes. Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with approximately 0.2 ml of methylene chloride.
- S. This is the concentrated PNA extract used below.

COLUMN CHROMATOGRAPHY CLEANUP

- A. A silica gel L.C. column is prepared by slurry packing 10 grams of silica gel (100-200 mesh) with methylene chloride into a 25 ml buret with teflon stopcock. A plug of glass wool is placed at the bottom of the column in order to prevent escape of the adsorbent during solvent elution.
- B. The LC column is washed with 50 ml of pentane prior to use.
- C. The concentrated sample extract is placed at the head of the column. Solvent is drained from the column so that the top of the extract just enters the column.
- D. The compounds of interest are eluted from the column with 25 ml of pentane followed by 40 ml of 40 percent (v/v) methylene chloride in pentane.
- E. The 40 percent (v/v) methylene chloride in pentane elutant is concentrated to dryness under a stream of nitrogen at 40°C.

F. The residual is redissolved in 1 ml of acetonitrite and transferred to a sealed amber colored sample vial. The sample is stored at 4°C until analysis.

ANALYSIS

SAMPLIA CONTINUE CONTINUE PROPER MANAGEM DO

ACASSOCIAL CONTRACTOR INCORPORA LOSSOCIALISMOS CONTRACTOR

Chromatograph conditions.

- A. Column: Perkin-Elmer 10u 0.26 x 25 cm HC-ODS
- B. Solvent program: 40 percent acetonitrite in water for 5 minutes programmed from 40-100 percent acetonitrite in 25 minutes
- C. Solvent flow rate: 1.0 ml/min
- D. Column temperature: 25°C
- E. Detector wavelengths:

UV #1: 254 nm UV #2: 300 nm

Fluorescence: Excitation: 220 nm Emission: 530 nm

F. Peaks are identified based on their retention time and on the comparison of the adsorbance at 254 nm and the fluorescence/absorbance at 254 nm ratios.

CALCULATION

- A. The recovery for each PNA in the water sample is determined by spiking previously extracted water samples with the standard stock solution of PNA's at a concentration of 5 µg/L each. The average recovery for each PNA is used as a correction factor.
- B. The quantitation of the PNA's is calculated by comparing the peak area of the water sample to the standard and using the correction factor.
- C. Analytical standards are injected each day to update the response factor.

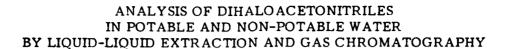
QUALITY ASSURANCE

Quality assurance is maintained by the following analytical routine:

- A. A laboratory water blank is analyzed to check for artifacts from the HPLC system and/or for the presence of impurities in the water blank making it unsuitable for standard preparation.
- B. The precision of the technique is determined by running 10 percent of the samples in duplicate.
- C. The accuracy of the technique is determined by spiking 10 percent of the samples and determining recoveries.

- D. The HPLC analyst proofreads the report and signs it on the bottom when satisfied that the results reported are val. 3.
- E. The final report is submitted to the senior organic chemist for final review. As a final check, the values reported are compared to the typical concentration range expected for the particular water supply.
- F. The worksheets, chromatograms, quantitation reports, etc. are fixed according to client by the lab clerk. The final report is duplicated and placed in the organics files, also according to client.
- G. The final report is sent out.

H. The remaining portion of the water sample is stored for one month at 4°C to allow reanalysis should there be any question of the results.



DEFINITION

The analysis of dihaloacetonitriles (DHAN's) consists of two parts: concentration by liquid-liquid extraction and analysis by gas chromatography (GC). The three dihalocetonitriles included in this are: dichloroacetonitrile (DCAN), chlorobromo-acetonitrile (CBAN), and dibromoacetonitrile (DBAN).

APPARATUS

The Varian 6000 gas chromatograph is a modular, dual column, dual detection instrument capable of isothermal or temperature programmed operation with packed or capillary columns and a variety of detectors.

REAGENTS

STATES STATES STATES

- A. Organic-free water is generated by passing deionized distilled water through a Barnstead Organic unit. This water is collected in a 20 liter bottle and heated to 60°C overnight. Contaminant free nitrogen is bubbled through the water at 50 ml/minute.
- B. Methanol ACS reagent grade
- C. Pentane-Pesticide Grade
- D. Standards:
 - 1. Dichloroacetonitrile
 - 2. Chlorobromoacetonitrile
 - 3. Dibromoacetonitrile
 - 4. 1,2-Dibromopropane (I.S.) ACS grade

STANDARD PREPARATION

A. Stock standard solutions

- 1. Fill two 50 ml glass stoppered volumetric flasks with approximately 49 ml of methyl alcohol. Let stand for a few minutes.
- 2. Add 7.3 µl of dichloroaceonitrile (10 mg) to one flask. Add 5.3 µl of chlorobromoacetonitrile (10 mg), and 4.4 µl of dibromoacetonitrile (10 mg) to the second flask.
- 3. Dilute to volume with methanol, stopper tightly, and invert the flasks three times. This produces stock standards at a concentration of 0.2 µg/µl.

NOTE: Stock standard is prepared on each day an analysis for dihalo-acetonitriles is to be performed.

B. Internal standard preparation

- 1. Fill a 50 ml glass stoppered, volumetric flask with approximately 45 ml of methanol. Let stand for a few minutes. Stopper the flask and weigh to +0.1 mg.
- 2. Add about 150 mg of standard to the flask immediately with syringe or pasteur pipet and reweigh.

NOTE: The standard drops should fall directly into the alcohol without contacting the flask neck.

- 3. Dilute to volume with methanol, stopper tightly and mix by inverting the flask several times.
- 4. Transfer approximately 1.5 ml into 2 ml vials. Seal with a TFE septum and screw cap, label and store the solution at 4°C. Calculate the concentration in µg per µl from the net gain in weight.
- 5. Determine the volume of concentrated standard in (4) needed to prepare a 1.724 mg/L solution.
- 6. Rinse a one liter flask three times with small volumes of pentane. Fill to volume with pentane, add the volume of concentrated internal standard determined in (5), stopper tightly and mix by inverting the flask two times.

EXTRACTION

A. Aqueous standard solutions for calibration

- 1. Three one liter and three 500 ml glass stoppered, volumetric flasks are filled to volume with laboratory organic free water and stoppered tightly.
- 2. Concentrations of 1, 2, and 5 µg/L of dichloroacetonitrile and concentrations of 10, 20, and 50 µg/L of chlorobromoacetonitrile and dibromoacetonitrile are prepared by injecting 5, 10, and 25 µl respectively of the dichloroacetonitrile stock standard and injecting 50, 100, and 250 µl respectively of the chlorobromoacetonitrile and dibromoacetonitrile stock standard into three one liter flasks.
- 3. Concentrations of 10, 20, and 50 µg/L of dichloroacetonitrile are prepared by injection of 25, 50, and 125 µl respectively of the dichloroacetonitrile stock standard into three 500 ml volumetric flasks.

- 4. The injection of stock standard solution is made by inserting the syringe needle well down into the bottom of the body of the flask, while holding the flask at a forty-five degree angle.
- 5. After addition of the standards, each flask is stoppered and slowly inverted three times to insure thorough mixing.
- 6. A 125 ml, amber, narrow-mouth bottle is filled with aqueous standards in excess of overflowing and sealed with TFE septa and screw caps. Invert the sealed bottles, tap and examine for the presence of bubbles. If bubbles are present, open bottles, add a few drops of standard and reseal.

B. Blank

AND THE CONTRACTOR CONTRACTOR

RESERVED CHARACTER TREATMENT TO SERVED AND SERVED CONTRACTOR OF THE PROPERTY O

- 1. The blank is the organic-free water used to prepare the standards.
- Fill two labelled 125 ml, amber, narrow-mouth bottles with organic-free water in excess of overflowing and seal with TFE teflon septa and screw cap. Check for the bubbles as with standard and sample preparation.

C. Extraction Procedure

NOTE: Prior to extracting any sample, test the extractant on the GC for impurities.

- Place each 125 ml amber bottle on a level surface. Remove the screw cap and TFE septum.
- 2. Using a 5 ml pipet with polypropylene disposable tips, remove 15 mls of sample from each bottle.
- 3. Add 30 grams of sodium sulfate to each bottle.
- Immediately add 5 ml of pentane-internal standard solution, recap, and invert bottle.
- 5. Let the bottles stand inverted on a level surface until they are ready to be placed on the shaker.
- 6. Shake the bottles on a gyrorotatory platform shaker for 20 minutes at 400 rpm, then place them on a level surface in the inverted position until extract collection.
- 7. Collect the extract with a disposable pasteur pipet and transfer into two 2 ml vials. Immediately seal the vials with a TFE septa, and screw caps. Label each vial.

8. Put one labeled vial in the carousel of the auto sampler of the Varian 6000 gas chromatograph for analysis. Store the second vial at 4°C for repeat analysis if required.

CHROMATOGRAPHIC ANALYSIS

The DHAN's are analyzed with the following instrument conditions:

Detector: Ni⁶³ electron capture Carrier gas: Ultrapure N₂ @ 80 PSI

Injector Pressure:

Split flow:

Makeup flow:

Column flow:

Injector temp.:

Detector temp.:

12 PSI

30 mls/min

30 mls/min

220°C

350°C

Column temp.: 40 degrees for three minutes, then temperature programmed at the rate of 10

perature programmed at the rate of 10 degrees per minute for two minutes, then 60 degrees is maintained for five

minutes.

Autosampler injection volume: 2 µl

Column type: Fused silica open tubular column 0.25

mm x 30 M coated with SE-54

CALCULATION

Contract Accounts

With the information developed from the standards and blanks develop a calibration curve for each of the dihaloacetonitriles. The calibration curve plots DHAN concentration versus integrated area counts. Area counts are normalized with the internal standard according to the following calculation:

Normalized DHAN Area Counts = (100,000) X (DHAN Area Counts)
Internal Standard Area Counts

QUALITY ASSURANCE

Analytical quality is assured by the following daily routine:

- A. Analyze extractant for impurities
- B. Analyze blank water for impurities
- C. Prepare and analyze standards in triplicate.
- D. Develop calibration curves for each of the DHAN's based on the average of the triplicate standards.
- E. Test precision by analyzing each twentieth sample in duplicate. If standard deviation is >10 percent, troubleshoot problems before continuing.

- F. Spike each twentieth sample with the DHAN's at between 1-20 ppb, check for accuracy. If accuracy is not ≤10 percent troubleshoot problems before continuing.
- G. Load autosampler with samples and standards (approximately one standard every five samples).
- H. Prepare a written report of sample DHAN concentrations and have it typed by the lab clerk. The lab clerk proofs the report for accuracy in typing. The analyst signs the report on the bottom when satisfied that the results reported are valid.
- I. The final DHAN report is submitted to the chief organic chemist for final review. This review includes a spotcheck of the chromatograms to ensure proper resolution and quantification.
- J. The worksheets, chromatograms, integrator values, and calibration curves are filed according to client by the lab clerk. The final report is duplicated and placed in the organic department's final report binder.
- K. The final report is sent to the client.

L. The duplicate DHAN extract is stored at 4°C for 30 days to allow reanalysis should there be any question of the results.

SECTION 2

PRECISION AND ACCURACY RESULTS

INORGANICS

NON-METALS (PHYSICAL/AESTHETIC, ANIONS, NUTRIENTS AND MAJOR CATIONS)

Two to five natural samples were analyzed in septuplet (7X) to determine precision at different levels.

Accuracy was determined as percent recovery on spikes added to the samples at different levels.

Detection limits are defined as three times the standard deviation of the lowest concentration sample.

or stressing a societies for each become representations of the each of the extent of the contractions and the contractions and the contractions are also the contractions ar

Key: \bar{x} = Average of replicates

THEOREM - CONTROL BELLEVILLE OF THE SECOND SERVICES CONTROL SECONDS

SD = Standard deviation

CV = Coefficient of variation

DISSOLVED OXYGEN (mg/L)

		Sample					
Replicate	A	<u>B</u>	<u>C</u>	<u>D</u>			
1	2.5	5.7	7.7	11.8			
2	2.4	5.7	7.8	11.9			
3	2.4	5.8	7.8	12.0			
4	2.5	5.8	7.8	11.8			
5	2.4	5.8	7.8	12.0			
6	2.4	5 . 7	7.7	11.9			
7	2.4	5.8	7.8	12.0			
×	2.4	5.8	7.8	11.9			
SD	0.05	0.05	0.05	0.09			
CV	2.1	0.9	0.6	0.8			

DL = 0.15 mg/L

TURBIDITY (NTU)

		Sample				
Replicate	A	B	_C			
1	5.5	35	3.0			
2	5.4	33	2.9			
3	5.1	34	2.8			
4	5.2	31	2.9			
5	5.7	32	2.8			
6	5.4	36	2.8			
7	5.4	35	3.0			
*	5.4	34	2.9			
SD	0.2	1.8	0.09			
CV	3.7	5.3	3.1			

TOTAL SUSPENDED SOLIDS (mg/L)

	San	nple
Replicate	A	B
1	17	74
2	18	76
3	19	82
4	19	76
5	16	74
6	19	82
7	17	82
ች	18	78
SD	1.2	3.8
CV	6.8	4.9

DL = 3.6

COLOR (in color units)

	Sample					
Replicate	A	В	C	_D		
1	35	40	5	5		
2	35	45	7	3		
3	25	40	5	3		
4	30	35	7	3		
5	25	45	7	2		
6	30	35	5	3		
7	30	45	5	2		
*	30	40.7	5.9	3.0		
SD	4.1	4.5	1.1	1.0		
cv	13.7	11.1	18.1	33.3		

DL = 3.0 color units

MBAS (mg/L LAS)

			Sample	2	
Replicate	<u>067</u>	038A + .10	<u>038A</u>	106A	106A + .15
1	.15	.21	.11	.075	.23
2	.15	.21	.09	.075	.23
3	.17	-21	.11	.075	.23
4	.13	-20	.12	.09	.21
5	.12	.18	.09	.06	.25
6	.13	.20	.09	.09	.23
7	.14	.18	.10	.065	.21
ž	.141	.199	.101	.076	.227
SD	.017	.013	.012	.011	.014
CV	11.8	6.8	8.2	15	6.0

% Recovery 038A + 0.10 = 99 106A + 0.15 = 100 DL = .033 mg/L

THE SECTION OF SECULAR SECTION OF SECULARIES SECTIONS SECULARIES.

CHLORINE RESIDUAL (mg/L)

	Sample						
Replicate	_ <u>A</u> _	В	<u>C</u>	D			
1	0.25	0.50	1.1	5.2			
2	0.25	0.50	1.0	5.1			
3	0.27	0.50	1.1	5.2			
4	0.24	0.60	1.1	5.1			
5	0.25	0.50	1.0	5.1			
6	0.27	0.60	1.0	5.2			
7	0.26	0.50	1.0	5.2			
ħ	0.26	0.53	1.0	5.2			
SD	0.01	0.05	0.05	0.05			
cv	3.9	9.4	4.8	1.0			
Known Conc.	0.25	0.50	1.0	5.0			

% Recovery

A = 104%

B = 106%

C = 104%

D = 103%

TOTAL DISSOLVED SOLIDS (mg/L)

		Sample					
Replicate	_ <u>A</u> _	В	C	D			
1	372	275	85	60			
2	372	278	78	54			
3	384	275	89	55			
4	364	249	89	52			
5	374	252	82	57			
6	364	234	82	61			
7	351	240	84	61			
*	369	258	84	57			
SD	10.3	18.2	4.4	3.6			
CV	2.8	7.1	5.2	6.3			

DL = 10.8 mg/L

Replicate	Sample					
	E	F	<u>G</u>	<u>H</u>		
1	49	100	245	496		
2	48	98	256	492		
3	48	97	246	490		
¥	48	98	249	493		
Known Conc.	50	100	250	500		
% Recovery	96.0	98.0	99.6	98.6		

ELECTRICAL CONDUCTIVITY (umhos/cm)

	Sample						
Replicate	A	B	C	D	E	F	
1	3.65	196	430	575	530	780	
2	3.30	195	435	580	530	780	
3	3.80	198	436	601	525	780	
4	3.72	197	435	599	530	778	
5	3.49	197	431	60 4	535	760	
6	3.20	195	438	603	530	770	
7	3.55	198	432	660	530	760	
X	3.53	197	434	594	530	773	
SD	0.22	1.27	2.91	12.74	2.89	9.29	
cv	6.2	0.7	0.7	2.2	0.5	1.2	
Known Conc.					528.3	770.6	

% Recovery

E = 100%

F = 100%

FLAME Ca (mg/L)

Replicate	<u>(1)</u>	(2)	<u>(5)</u>	Sample		
<u> </u>	<u> </u>	<u>,</u>	<u>(3)</u>	<u>(4)</u>		No Spike
Expected	5.3	4.3	8.3	13.3	23.3	
1	4.9	4.2	8.0	13.1	24.4	3.3
2	4.9	4.1	8.4	13.7	24.2	3.3
3	4.9	4.1	7.8	12.8	24.6	3.2
4	4.9	4.1	8.0	12.9	23.5	3.4
5	4.9	4.0	8.0	13.1	24.1	3.3
6	4.9	4.2	8.0	13.1	24.0	3.4
7	5.0	4.1	7.9	13.1	24.3	3.3
ž	4.9	4.1	8.0	13.1	24.2	3.3
SD	.038	.069	.186	.285	.351	
cv	0.77	1.68	2.33	2.18	1.45	
Percent Recovery	92	95	96	98	104	

FLAME Mg (mg/L)

			<u>Spike</u>			
Replicate	<u>(1)</u>	<u>(2)</u>	<u>(3)</u>	<u>(4)</u>	<u>(5)</u>	Sample
						No Spike
Expected	1.9	2.9	3.4	5.9	10.9	
1	1.8	2.7	3.3	5.7	10.6	0.8
2	1.8	2.7	3.2	5.7	10.7	0.9
3	1.7	2.7	3.2	5.7	10.6	0.9
4	1.8	2.6	3.2	5.6	10.6	0.9
5	1.8	2.7	3.2	5.7	10.6	0.8
6	1.8	2.7	3.2	5.6	10.8	0.9
7	1.8	2.7	3.2	5.8	10.7	0.9
x	1.8	2.7	3.2	5.7	10.7	0.9
SD	.038	.038	.038	0.069	0.079	
cv	2.12	1.41	1.18	1.21	0.74	5.60
Percent Recovery	95	93	94	97	98	

FLAME K (mg/L)

			Spike			
Replicate	<u>(1)</u>	<u>(2)</u>	<u>(3)</u>	<u>(4)</u>	<u>(5)</u>	Sample
						No Spike
	2.1	4.1	. 1	10.1	20.1	
Expected	2.1	4.1	5.1	10.1	20.1	
1	1.8	3.9	4.9	10.0	20.4	0.0
2	1.8	3.9	4.9	10.1	20.4	0.1
3	1.9	3.9	4.9	10.1	20.6	0.1
4	1.9	3.9	4.9	10.0	20.3	0.0
5	2.0	3.9	4.9	9.9	20.4	0.0
6	2.0	3.9	4.9	10.0	20.3	0.0
7	1.9	3.9	4.9	10.0	20.5	0.0
ž	1.9	3.9	4.9	10.0	20.4	0.1
SD	.082	0.000	0.000	.069	.107	
cv	4.30	0.00	0.00	0.69	0.52	
Percent				·		
Recovery	90	95	96	99	101	

FLAME Na (mg/L)

			Spike			
Replicate	<u>(1)</u>	(2)	<u>(3)</u>	<u>(4)</u>	<u>(5)</u>	Sample
						No Spike
Expected	3.6	6.6	8.6	15.6	30.6	
•	2.4	4.0	0.0	1/ /	22.0	0.7
1	3.4	6.8	8.3	16.6	32.3	0.6
2	3.4	6.7	8.3	16.5	31.9	0.6
3	3.4	6.7	8.2	16.3	32.1	0.6
4	3.4	6.7	8.3	16.5	32.6	0.6
5	3.5	6.7	8.3	16.6	32.0	0.6
6	3.4	6.7	8.4	16.6	31.7	0.6
7	3.4	6.6	8.4	15.5	31.5	0.6
ž	3.4	6.7	8.3	16.4	32.0	0.6
SD	0.038	.058	.069	.399	0.367	0.00
cv	1.11	0.86	0.83	2.44	1.15	0.00
Percent Recovery	94	102	97	105	105	

DL = 0.1

THE COURSE THE STANDARD STANDA

ALKALINITY (mg/L CaC0₃)

		<u>Sample</u>						
Replicate	<u>156A</u>	<u>257</u>	<u>315</u>	156A + 30	315 + 90			
1	18.3	65	160	47.8	246			
2	18.3	68.3	160	46.6	247.5			
3	16.6	65	160	45.5	246			
4	16.6	66.6	160	45.5	249			
5	18.3	68.3	161.6	45.5	247.5			
6	18.3	66.6	161.6	45.4	244.5			
7	16.8	66.5	161.6	43.4	246			
*	17.6	66.6	160.6	45.6	246.6			
SD	.87	1.3	.85	1.3	1.46			
cv	4.9	2.0	0.5	2.9	0.6			

% Recovery

156A + 30 = 96%

315 + 90 = 99%

DL = 2.7 mg/L

BROMIDE (mg/L Br)

Sample

					+.1	+1.0
Replicate	006-1	006-2	<u>446</u>	446-1	006-2	446-1
1	.050	.066	.091	.029	.162	1.02
2	.048	.065	.093	.026	.153	.995
3	.053	.065	.097	.026	.153	1.00
4	.048	.065	.093	.027	.159	.987
5	.048	.063	.096	.026	.166	.983
6	.049	.064	.093	.027	.145	.982
7	.052	.065	.099	.028	.158	.971

.095

.0028

3.0

.027

.001

4.3

.991

.016

1.6

.157

.007

4.4

% Recovery
446-1 + 1 ppm = 97%
006-2 + 0.1 ppm = 95%
DL = .003 mg/L

.065

.001

1.4

.050

.002

4.1

Ā

SD CV

CHLORIDE (mg/L Cl) (0-50 ppm)

Replicate	315	<u>156</u>	<u>154</u>	<u>242</u>	<u>156 + 10</u>	<u>315 + 20</u>
_	24.0	4.0	1 20	20.0	14 5	44.75
1	26.0	4.0	1.30	30.0	14.5	44.75
2	26.1	3.8	1.30	29.0	14.5	44.5
3	26.0	3.8	1.28	32.0	14.8	45.0
4	26.5	3.8	1.26	29.0	14.0	46.09
5	26.5	4.0	1.21	30.5	14.0	45.01
6	26.5	3.9	1.24	30.3	14.0	44.5
7	26.0	3.8	1.30	29.5	14.5	
X	26.23	3.8	1.27	30.04	14.3	44.97
SD	.26	.095	.035	1.04	0.32	.59
cv	0.98	2.4	2.7	3.4	2.27	1.31

% Recovery

156 + 10 = 104%

315 + 20 = 97%

DL = 0.11 mg/L

CN (ug/L CN)

			Sample		
	+10 ug/L		+60 ug/L		
Replicate	373A	<u>373B</u>	373B	373A	<u>373C</u>
1	19.9	41.7	99.3	13.8	79.2
2	23.8	41.6	97.9	13.6	79.9
3	22.6	40.6	100.0	15.9	79.1
4	24.8	42.9	103.7	17.2	82.8
5	26.5	45.3	101.0	16.9	83.6
6	26.6	45.2	101.7	18.3	81.7
7	27.1	44.3	102.3	15.7	84.9
x	24.5	43.1	100.8	15.9	81.6
SD	2.58	1.88	1.95	1.72	2.29
cv	10.5	4.0	2.3	10.8	3.7

% Recovery

373A + 10 ppb = 94%

373B + 60 ppb = 98%

DL = 5 ug/L

FLUORIDE (mg/L F)

	Sample					
Replicate	369 + .8	342	342 + .2	341	<u>369</u>	Tap.
1	1.60	.58	.80	.40	.80	.55
2	1.62	.62	.75	.38	.85	.52
3	1.70	•58	.76	.45	.80	.55
4	1.66	.62	.76	.38	.80	.56
5	1.7	.62	.80	.44	.82	.58
6	1.65	.50	.80	.44	.80	.51
7	1.70	.60	.78	.36	.85	.55
x	1.66	.58	.77	.40	.817	.545
SD	.0409	.043	.022	.036	.023	.023
cv	2.46	7.3	2.8	8.8	2.8	4.3

% Recovery

369 + .8 = 97%

342 + .2 = 99%

DL = .07 ppm

IODIDE (ug/L)

	Sample					
			A +		B +	
Replicate	75509	_ <u>A</u> _	.5 ppb	<u>B</u>	5 ppb	
1	4	10	12	12	17	
1	4	10	12	12	17	
2	4	10	11	12	18	
3	4	10	12	12	20	
4	4	10	12	12	19	
5	5	10	14		19	
x	4.2	10	12.2	12	18.6	
SD	.45	0	1.1	0	1.1	
cv	10	0	8.9	0	6.1	

% Recovery
B + 5 ug/L = 109%
A + 2.5 ug/L = 98%
DL = 1.5 ug/L

N0₃ (mg/L N)

				Sample			
Replicate	209	<u>156</u>	208	249	249 + .5	243	<u>243 + .3</u>
1	.440	.743	1.52	-948	1.41	.083	.434
2	.441	.747	1.50	.942	1.42	.083	.415
3	.430	.737	1.54	.947	1.41	.093	.413
4	.440	.741	1.46	.945	1.43	.083	.414
5	.453	.734	1.45	.938	1.41	.093	.375
6	.458	.740	1.48	.944	1.45	.093	.375
7	.448	.755	1.47	.946	1.45	-093	.375
Ā	.444	.742	1.48	.944	1.42	.088	.400
SD	.0093	.0069	.032	0.34	.018	.0053	.024
cv	2.1	0.93	2.2	0.36	1.27	6.0	6.1

% Recovery

249 + .5 = 101

243 + .3 = 97

DL = .015 mg/L N

AMMONIA (mg/L NH₃)

			Sample	<u>!</u>	
Replicate	102+.1	103	103+.5	102	101
1	.252	1.002	1.48	.141	.116
2	.215	1.007	1.47	.150	.116
3	.253	1.014	1.50	.153	.115
4	.262	1.007	1.49	.158	.125
5	.246	1.008	1.500	.141	.125
6	.252	1.009	1.516	.158	.120
7	.232	1.004	1.47		.125
ž	.244	1.007	1.49	.150	.120
SD	.0159	.0038	1.57	.0077	.0046
CV	6.5	0.37	1.05	5.14	3.8

% Recovery

103 + .5 = 99%

102 + .1 = 98%

DL = 0.014 mg/L

TOTAL KJELDAHL NITROGEN (mg/L - N)

			Sample	<u> </u>	
Replicate	<u>193</u>	193+1	194	4	4+.2
1	.543	1.69	1.72	.267	.520
2	.591	1.72	1.73	.458	.516
3	.617	1.81	1.75	.321	.542
4	.634	1.89	1.85	.449	.660
5	.697	1.77	1.83	.414	.559
6	.701	2.57	1.80	.424	.645
7	.594	1.68	1.84	.360	.611
₹	.625	1.76	1.78	.384	.579
SD	.0577	.080	.054	.071	•059
CV	9.2	4.5	3.0	18.5	10.2

% Recovery

193 + 1 = 108%

4 + .2 = 99%

DL = .21 mg/L

PHOSPHATE (mg/L P)

		Sample					
Replicate	<u>395</u>	<u>396</u>	396A	396A + .1	<u>395A</u>	395 + .3	
1	.451	.573		.124	.0509	.7407	
2	.482	.555	.0635	.141	.0549	.7479	
3	.434	.543	.0604	.138	.0587	.750	
4	.478	.527	.0611	.145	.0407	.722	
5	.495	.553	.0617	.154	.0546	.7885	
6	.483	.589	.0550	.161	.0553	.7240	
7	.491	.609	.0664	.148	.0557	.7456	
x	.473	.564	.0614	.144	.0529	.745	
SD	.022	.028	.0036	.0119	.0058	.021	
cv	4.7	4.9	5.8	8.2	11.0	2.9	

% Recovery

395 + 0.3 ppm = 96%

396A + .1 ppm = 89%

DL = 0.011 mg/L

SILICA (mg/L SiO₂)

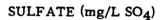
	Sample					
		75723		75728		
Replicate	75723	+3.6 ppm	75728	+.6 ppm	75447	
1	6.3	9.3	4.0	4.6	8.1	
2	6.2	9.3	3.9	4.4	8.1	
3	6.3	9.3	3.9	4.6	8.1	
4	5.9	9.4	3.8	4.6	7.9	
5	5.9	9.4	3.9	4.3	8.0	
*	6.1	9.3	3.9	4.5	8.0	
SD	0.2	0.05	0.07	0.14	0.09	
cv	3.3	.5	1.8	3.1	1.1	

% Recovery

75723 + 3.6 = 96%

75728 + .6 = 100%

DL = 0.2 mg/L



	Sample							
Replicate	322	312+10	227	322+20	311	312	200	<u>313</u>
1	23.0	22.6	28.5	39.6	15.1	12.9	38.2	40.6
2	23.8	22.6	29.6	39.8	15.2	13.3	37.8	39.3
3	24.3	22.6	29.8	39.8	15.3	13.0	37.7	39.3
4	25.0	24.4	29.2	39.5	15.1	13.0	38.1	39.5
5	24.2	22.1	28.6	41.9	15.6	13.3	38.1	38.3
6	24.8	23.7	30.1	41.0	14.9	12.7	37.4	38.0
7	24.8		31.4	39.8	14.5	12.8	37.7	37.0
₹	24.3	23.0	29.6	40.2	15.1	13.0	37.9	38.9
SD	.7	.8	•9	•9	.3	.2	.2	1.1
cv	2.9	3.7	3.3	2.2	2.2	1.6	.74	2.8

% Recovery

322 + 20 mg/L = 91%

312 + 10 mg/L = 100%

DL = .6 mg/L

ASBESTOS REPLICATES FROM SINGLE SAMPLE SITE

Replicate	MFL*	Mass (ug/L)
1	111.04	0.54
2	123.93	0.58
3	102.12	0.36
4	101.13	0.51
5	113.02	0.33
6	85.66	0.50
7	68.08	0.16
Mean	100.71	0.42
SD	18.67	0.15
%SD	18.53	35.04

^{*} Concentrations in millions of fibers/liter

ASBESTOS REPLICATE SAMPLE GRIDS PREPARED FROM SINGLE FILTER

MFL*	Mass (ug/L)
47	1.98
36	2.30
36	1.67
43	1.90
42	2.15
34	1.77
39	2.70
39.57	2.06
4.64	0.35
11.74	17.00
	47 36 36 43 42 34 39

^{*} Concentrations in millions of fibers/liter

THE THE PROPERTY OF THE PROPERTY OF THE PROPERTY AND THE PROPERTY OF THE PROPE

METALS

A natural sample low in metals was spiked at five different concentrations and the precision determined by septuplet analyses at each concentration level. All values are in mg/L.

Accuracy is based on comparison of theoretical results with the average value for each spiked concentration.

Detection limits are calculated as three times S.D. for the lowest concentration sample. The practical detection limit is five to seven times lower for total metals since the digestion method results in a concentration factor based on digesting a volume of 100 cc to 15 cc final volume.

FURNACE Ag

			<u>Spi</u>	<u>ke</u>		
Replicate	20 ppb	10 ppb	5 ppb	2 ppb	1 ppb	
1	18.7	9.5	4.8	2.0	0.9	0.1
2	20.3	9.3	4.8	2.1	1.0	0.2
3	20.3	9.3	4.9	1.9	1.4	0.0
4	20.9	9.3	4.7	2.6	1.0	0.2
5	20.4	9.3	4.8	1.9	1.0	0.0
6	19.1	9.0	4.9	2.1	0.7	0.4
7	17.4	9.0	4.8	2.1	0.8	0.0
*	19.6	9.2	4.8	2.1	0.97	.13
SD	1.24	.18	.07	.24	.22	.15
cv	6.3	2.0	1.4	11.3	22.8	116
(x - 0)	19.5	9.1	4.7	2.0	.84	0
Percent Recovery	98	91	94	100	84	

DL = 0.7 ppb

FLAME Ag (mg/L)

			Spike		
Replicate	<u>(1)</u>	<u>(2)</u>	<u>(3)</u>	<u>(4)</u>	<u>(5)</u>
Expected	.010	.100	.500	2.000	.020
1	.013	.097	.489	1.994	.019
2	.011	.100	.489	1.989	.020
3	.011	.099	.4 89	1.990	.022
4	.008	.102	.504	1.975	.023
5	.012	.098	.503	1.995	.019
6	.011	.101	.4 98	1.990	.020
7	.012	.101	.498	1.998	.020
₹	.0111	.0997	.4957	1.9901	.0204
SD	.0016	.0015	.0018	.0067	.0074
Percent Recovery	111	100	99	100	102

ICP Al (mg/L)

			Spike		
Replicate	<u>(1)</u>	<u>(2)</u>	(3)	<u>(4)</u>	<u>(5)</u>
Expected	.100	.500	1.000	2.000	.200
1	.096	.512	1.046	2.003	.196
2	.092	.503	1.003	1.986	.190
3	.098	.486	.963	1.972	.184
4	.091	.487	.989	1.982	.189
5	.108	.482	.994	1.993	.201
6	.089	.469	.982	1.989	.194
7	.090	.491	1.008	2.005	.191
₹	.0949	.490	.998	1.990	.192
SD	.0066	.0140	.0258	.0116	.0055
cv	7.0	2.9	2.6	.6	2.8
Percent Recovery	95	98	100	100	96

gare inamen vancen ikkeske ingeren proesa ekekse mann weren presen waren bi

FURNACE As (mg/L)

20 ul +20 ul Ni(NO₃)₂

			Spike		
Replicate	<u>(1)</u>	<u>(2)</u>	<u>(3)</u>	<u>(4)</u>	<u>(5)</u>
Expected	.005	.020	.050	.100	.010
1 .	.0053	.0190	.0492	.1014	.0108
2 ·	.0054	.0202	.0503	.1003	.0104
3	.0052	.0203	.0511	.0992	.0102
4	.0045	.0205	.0493	.1014	.0098
5	.0055	.0205	.0493	.1003	.0105
6	.0048	.0201	.0490	.1012	.0101
7	.0053	.0197	.0481	.1009	.0102
#	.0051	.0200	.0495	.1007	.0103
SD	.00036	.00054	.00096	.00080	.00032
cv	7.0	2.7	1.9	0.79	3.1
Percent Recovery	102	100	99	101	103

ICP B (mg/L)

			Spike		
Replicate	<u>(1)</u>	<u>(2)</u>	<u>(3)</u>	<u>(4)</u>	<u>(5)</u>
Expected	.020	.040	.080	.200	1.00
1	.014	.044	.089	.235	1.002
2	.036	.052	.092	.214	1.049
3	.022	.048	.081	.220	1.063
4	.016	.036	.072	.223	1.032
5	.029	.038	.083	.223	1.008
6	.018	.032	.090	.207	1.016
7	.034	.046	.080	.196	1.021
x	.0241	.0421	.0839	.2169	1.027
SD	.0088	.0071	.0070	.0126	.0222
cv	36.8	16.8	8.4	5.8	2.2
Percent Recovery	121	105	105	108	103

ICP Ba (mg/L)

			Spike		
Replicate	<u>(1)</u>	<u>(2)</u>	(3)	<u>(4)</u>	<u>(5)</u>
Expected	.010	.020	.040	.500	2.000
1	.008	.017	.045	.508	2.033
2	.015	.019	.042	.522	2.017
3	.016	.025	.046	.533	2.053
4	.013	.026	.048	.509	2.041
5	.009	.022	.041	.498	2.009
6	.006	.024	.036	.509	2.042
7	.008	.020	.039	.511	2.013
#	.0107	.0218	.0424	.5129	2.030
SD	.0039	.0033	.0042	.0113	.0168
cv	36.4	15.3	9.9	2.2	.8
Percent Recovery	107	109	106	103	102

ICP Be (mg/L)

			Spike	
Replicate	<u>(1)</u>	<u>(2)</u>	(3)	<u>(4)</u>
Expected	.050	.200	0.100	.020
1	.053	.233	.124	.022
2	.053	.222	.120	.020
3	.053	.228	.119	.021
4	.054	.234	.121	.019
5	.053	.228	.117	.022
6	.052	.231	.122	.022
7	.052	.241	.121	.021
₹	.053	.231	.120	.021
SD	.0009	.0057	.0022	.0012
cv	1.7	2.5	1.9	5.6
Percent Recovery	106	116	120	105

FURNACE Cd (mg/L)

			Spike		
Replicate	<u>(1)</u>	<u>(2)</u>	<u>(3)</u>	<u>(4)</u>	<u>(5)</u>
Expected	.100	.050	.020	.010	.005
1	.106	.052	.021	.013	.006
2	.090	.052	.020	.012	.006
3	.095	.051	.020	.013	.006
4	.098	.054	.021	.013	.006
5	.108	.054	.022	.012	.005
6	.097	.048	.018	.012	.006
7	.102	.053	.020	.012	.006
Æ	.099	.052	.020	.012	.006
SD	.0063	.0021	.0013	.0005	.0004
cv	6.4	4.0	6.2	4.3	6.5
Percent Recovery	99	104	100	120	120

ICP Co (mg/L)

			Spike		
Replicate	<u>(1)</u>	<u>(2)</u>	(3)	<u>(4)</u>	<u>(5)</u>
Expected	.025	.2	•5	2.000	.050
1	.040	.204	.682	2.042	.060
2	.028	.187	.643	1.976	.069
3	.037	.192	.644	1.974	.065
4	.024	.180	.628	1.869	.064
5	.036	.215	.643	1.910	.071
6	.016	.201	.615	1.910	.053
7	.026	.178	.622	1.915	.044
₹	.029	.194	.640	1.942	.0609
SD	.0085	.0136	.0220	.0584	.0095
cv	29.0	7.0	3.4	3.0	15.6
Percent Recovery	116	97	128	97	122

NON VETERIOR DISERBOR PROPERTY NOTITION SECRETAR EXCRACL BURGARD DESCRIPTION OF THE PARTY OF THE PARTY NAMED OF THE PARTY NAMED

FURNACE Co (ppb)

	<u>Spike</u>					
Replicate	20 ppb	10 ppb	5 ppb	2 ppb	1 ppb	
						_
1	21.5	11.5	5.9	2.4	2.1	0.4
2	21.1	11.1	5.6	2.5	1.8	0.4
3	21.5	11.0	5.9	2.8	1.8	0.3
4	21.3	11.2	5.8	2.6	1.9	0.5
5	20.7	11.3	6.0	2.6	1.6	0.6
6	21.6	10.9	5.9	2.7	1.6	0.3
7	21.0	11.1	5.8	2.8	1.7	0.6
7.	21.2	11.2	5.8	2.6	1.8	.44
SD	0.33	.20	.127	.150	.177	.127
cv	1.5	1.8	2.2	5.7	9.9	28.7
(% - 0)	20.8	10.8	5.4	2.2	1.4	0
Percent Recovery	104	108	108	110	140	

DL = 0.5 ppb

FURNACE Cr (mg/L)

			Spike		
Replicate	<u>(1)</u>	<u>(2)</u>	(3)	<u>(4)</u>	<u>(5)</u>
Expected	.005	.010	.020	.050	.100
1	.005	.010	.021	.048	.100
2	.004	.010	.022	.047	.103
3	.005	.010	.021	.048	.100
4	.005	.010	.020	.050	.103
5	.005	.011	.021	.048	.102
6	.005	.010	.021	.047	.103
7	.005	.010	.019	.049	.101
₹	.005	.010	.021	.048	.102
SD	.00038	.00038	.00095	.0011	.0014
cv	7.8	3.7	4.6	2.2	1.4
Percent Recovery	100	101	105	96	102

ICP Cr (mg/L)

Replicate	<u>(1)</u>	<u>(2)</u>	Spike (3)	<u>(4)</u>	<u>(5)</u>
Replicate	(1)	<u>(L)</u>	(3)	<u>(4)</u>	(3)
Expected	.025	.200	.500	2.000	.050
1	.033	.239	.523	2.334	.037
2	.009	.203	.536	2.190	.025
3	.025	.189	.571	2.311	.027
4	.017	.194	.548	2.362	.037
5	.023	.201	.556	2.264	.042
6	.019	.236	.571	2.125	.054
7	.017	.219	.543	2.150	.046
#	.0204	.2116	.5497	2.2480	.0383
SD	.0075	.0200	.0178	.0937	.0102
Percent Recovery	82	106	110	112	77

FLAME Cu (mg/L)

			Spike		
Replicate	<u>(1)</u>	<u>(2)</u>	<u>(3)</u>	<u>(4)</u>	<u>(5)</u>
Expected	.010	.020	.100	.500	2.000
1	.021	.029	.089	.469	1.874
2	.018	.026	.100	.468	1.871
3	.021	.032	.095	.462	1.898
4	.019	.038	.106	.458	1.874
5	.024	.029	.101	.459	1.897
6	.017	.028	.096	.474	1.861
7	.020	.030	.099	.461	1.878
₹	.0200	.0303	.0980	.4644	1.879
SD	.0023	.00386	.0054	.0060	.0137
cv	11.55	12.75	5.46	1.29	.73
Percent Recovery	200	152	98	93	94

ICP Cu (mg/L)

			Spike		
Replicate	<u>(1)</u>	<u>(2)</u>	<u>(3)</u>	<u>(4)</u>	<u>(5)</u>
Expected	.050	.100	.200	.500	2.000
1	.050	.103	.204	.489	1.975
2	.053	.103	.197	.487	1.982
3	.051	.105	.198	.496	1.972
4	.052	.103	.208	.508	1.984
5	.048	.104	.206	.491	1.996
6	.053	.102	.205	.493	1.984
7	.052	.105	.209	.489	1.979
Ī	.0513	.104	.204	.493	1.982
SD	.0018	.0011	.0047	.0071	.0077
cv	3.5	1.1	2.3	1.4	0.4
Percent Recovery	103	104	102	99	99



ICP Fe (mg/L)

			Spike		
Replicate	<u>(1)</u>	<u>(2)</u>	(3)	<u>(4)</u>	<u>(5)</u>
Expected	.025	.200	.500	4.000	.050
1	.018	.216	.515	4.001	.058
2	.025	.204	.531	4.014	.042
3	.027	.212	.499	4.011	.053
4	.033	.201	.524	3.993	.043
5	.020	.196	•509	3.989	.050
6	.029	.205	.514	3.998	.049
7	.027	.212	.528	4.003	.052
ž	.0256	.2066	.5171	4.001	.0496
SD	.0052	.0071	.0113	.0090	.0056
cv	20.2	3.4	2.2	0.2	11.3
Percent Recovery	102	103	103	100	99

DL = .016



THE THIRD SESSION CONTROL LESSESSE SECTION SECTION SECTION SECTION OF SECTION

Hg (ug/L Hg by Coleman Analyzer)

Replicate	Sample 1	1+0.5 ug/L	1+1.0 ug/L	1+2.0 ug/L	Sample 2
1	1.12	1.70	2.45	3.50	.40
2	1.40	1.78	2.42	3.20	.18
3	1.25	1.69	2.20	3.25	.28
4	1.27	1.69	2.24	3.30	.29
6	1.22	1.76	2.23	3.15	.24
7	1.18	1.69	2.15	3.20	.24
8	1.19	1.89	2.30	3.30	.33
Ave	1.23	1.73	2.28	3.27	.28
SD	•09	.05	.11	.11	.07
cv	7.2	2.7	4.9	3.5	25.0

DL = 0.27 ug/L

% Recovery

Sample 1 + 1 = 102%

Sample 1 + .5 = 100%

FLAME Li (mg/L)

				<u>Spike</u>			
							Blank
Replicate	<u>(1)</u>	<u>(2)</u>	<u>(3)</u>	<u>(4)</u>	<u>(5)</u>	<u>(6)</u>	Sample
Expected	.015	.025	.055	.105	.505	1.005	.005
1	.014	.024	.055	.110	.509	1.020	.005
2	.014	.025	.055	.110	.509	1.023	.004
3	.016	.027	.054	.107	.513	1.014	.006
4	.014	.024	.058	.108	.516	1.014	.004
5	.014	.027	.057	.111	.511	1.022	.003
6	.015	.026	.057	.111	.509	1.017	.008
7	.014	.028	.058	.111	.512	1.014	.005
ž	.014	.026	.056	.110	.511	1.018	.005
SD	.0008	.0016	.0016	.0016	.0026	.0039	.0016
cv	5.5	6.1	2.9	1.5	0.5	0.4	32.7
Percent Recovery	93	104	102	105	101	101	

DL = .002

STANKER SERVICES IN SUBMINIST PRESENTED WITHOUT

ICP Li (mg/L)

				Spike		
						Blank
Replicate	<u>(1)</u>	<u>(2)</u>	<u>(3)</u>	<u>(4)</u>	<u>(5)</u>	Sample
Expected	.015	.025	.055	.105	.505	
1	•009	.022	.055	.106	.506	.000
2	.011	.023	.053	.105	.514	.001
3	.011	.022	.054	.107	.506	.000
4	.010	.021	.054	.105	.513	.000
5	.008	.021	.054	.101	.504	.001
6	.011	.022	.054	.104	.490	.001
7	.013	.021	.053	.104	.491	.000
*	.010	.022	.054	.105	.503	.000
SD	.0016	.0007	.0007	.0019	.0096	.0007
CV	15.7	3.1	1.4	1.8	1.9	483.1
Percent Recovery	67	88	98	100	100	

ICP Mn (mg/L)

			Spike		
Replicate	<u>(1)</u>	<u>(2)</u>	(3)	<u>(4)</u>	<u>(5)</u>
Expected	.010	0.100	0.500	2.000	0.020
1	.012	.112	.489	2.074	.018
2	.009	.108	.49 6	2.062	.022
3	.009	.105	.492	2.068	.021
4	.008	.109	.513	2.076	.017
5	.010	.098	.496	2.081	.021
6	.013	.097	.508	2.071	.020
7	.008	.104	.521	2.080	.020
₹	.0099	.1047	.5021	2.073	.0199
SD	.0020	.0056	.0120	.0067	.0018
cv	19.8	5.3	2.4	3.3	8.9
Percent Recovery	99	105	100	104	100

ICP Mo (mg/L)

			Spike		
Replicate	<u>(1)</u>	<u>(2)</u>	<u>(3)</u>	<u>(4)</u>	<u>(5)</u>
Expected	.040	.100	.500	1.000	4.000
1	.048	.112	.486	1.028	4.092
2	.042	.108	.522	1.074	4.067
3	.046	.114	.512	1.039	4.014
4	.038	.110	.508	1.002	4.038
5	.041	.102	.510	1.036	4.072
6	.045	.108	.502	1.028	4.014
7	.043	.096	.524	1.022	4.016
#	.043	.107	.509	1.033	4.045
SD	.0034	.0062	.0128	.0218	.0322
CV	7.7	5.8	2.5	2.1	0.8
Percent Recovery	108	107	102	103	101

ICP Ni (mg/L)

			Spike		
Replicate	<u>(1)</u>	<u>(2)</u>	<u>(3)</u>	<u>(4)</u>	<u>(5)</u>
Expected	.05	.10	.20	.50	1.00
1	.056	.102	.218	.542	1.089
2	.053	.116	.216	.506	1.067
3	.050	.108	.202	.519	1.052
4	.053	.109	.197	.524	1.062
5	.055	.100	.196	.536	1.029
6	.054	.096	.224	.521	1.043
7	.058	.108	.218	.518	1.038
z	.054	.1056	.210	.524	1.054
SD	.0025	.0067	.0115	.0120	.0203
cv	4.7	6.3	5.5	2.3	1.9
Percent Recovery	108	106	105	105	105

DL = .008

FURNACE Pb (mg/L)

Replicate	<u>(1)</u>	<u>(2)</u>	<u>Spike</u> (3)	<u>(4)</u>	<u>(5)</u>
Expected	.010	.020	.050	.100	.200
-					
1	.010	.020	.054	.095	.194
2	.010	.020	.050	.099	.191
3	.009	.019	.051	.110	.187
4	.009	.021	.052	.099	.208
5	.009	.021	.051	.096	.195
6	.009	.021	.048	.098	.200
7	.009	.021	.051	.104	.206
*	.0093	.020	.051	.100	.197
SD	.00049	.00079	.0018	.0052	.0077
cv	5.3	3.9	3.6	5.2	3.9
Percent Recovery	93	100	102	100	99

FURNACE Sb (mg/L)

			Spike		
Replicate	<u>(1)</u>	<u>(2)</u>	(3)	<u>(4)</u>	<u>(5)</u>
Expected	.010	.020	.040	.100	.250
1	.013	.021	.039	.105	.252
2	.014	.021	.040	.103	.244
3	.013	.021	.038	.103	.247
4	.014	.020	.040	.101	.251
5	.013	.021	.040	.104	.250
6	.013	.021	.039	.107	.252
7	.013	.021	.040	.103	.251
₹	.013	.021	.039	.104	.250
SD	.00049	.00038	.00079	.00190	.0030
cv	11.0	1.8	2.0	1.8	1.2
Percent Recovery	130	105	98	104	100

DL = .0015

CONTRACT RECESSORY (SECRETARY LANGUAGE) (SECRETARY SECRETARY)

FURNACE Se (mg/L) (20 ul + 20 ul Ni(NO₃)₂)

			Spike		
Replicate	<u>(1)</u>	<u>(2)</u>	<u>(3)</u>	<u>(4)</u>	<u>(5)</u>
Expected	.005	.020	.050	.100	.010
1	.0052	.0217	.0525	.1019	.0104
2	.0049	.0210	.0514	.1009	.0107
3	.0053	.0210	.0530	.1035	.0103
4	.0046	.0214	.0515	.1034	.0105
5	.0048	.0214	.0514	.1006	.0104
6	.0052	.0210	.0518	.0991	.0105
7	.0052	.0205	.0518	.0979	.0102
#	.0050	.0211	.0519	.1010	₀ 010 4
SD	.00026	.00039	.00061	.0021	.00016
cv	5.2	1.8	1.2	2.1	1.5
Percent Recovery	100	106	104	101	104

ICP Sn (mg/L)

			Spike		
Replicate	<u>(1)</u>	<u>(2)</u>	(3)	<u>(4)</u>	<u>(5)</u>
Expected	.100	.200	0.500	2.500	10.000
1	.092	.218	.489	2.527	10.389
2	.112	.223	.523	2.462	10.162
3	.104	.214	.525	2.617	10.083
4	.108	.200	.524	2.549	10.042
5	.112	.214	.509	2.610	10.069
6	.109	.223	.518	2.503	10.003
7	.111	.203	.520	2.496	10.096
Æ	.1069	.214	.515	2.538	10.121
SD	.0071	.0091	.0128	.058	.1280
cv	6.7	4.2	2.5	2.3	1.3
Percent Recovery	107	107	103	102	101

FURNACE Tl (mg/L)

			Spike		
Replicate	<u>(1)</u>	<u>(2)</u>	<u>(3)</u>	<u>(4)</u>	<u>(5)</u>
Expected	.01	.025	.05	.1	.2
1	.012	.024	.054	.099	.214
2	.014	.026	.053	.099	.216
3	.010	.028	.056	.097	.202
4	.010	.027	.052	.102	.214
5	.011	.023	.052	.110	.198
6	.009	.025	.054	.104	.208
7	.009	.024	.054	.106	.204
Ā	.011	.025	.054	.102	.208
SD	.0018	.0018	.0014	.0046	.0069
cv	16.8	7.1	2.6	4.5	3.33
Percent Recovery	110	100	108	102	104



Replicate	<u>(1)</u>	<u>(2)</u>	<u>Spike</u> (3)	<u>(4)</u>	<u>(5)</u>
Expected	.020	.050	.100	.200	1.000
1	.016	.049	.102	.204	1.022
2	.026	.056	.110	.214	0.982
3	.014	.043	.091	.219	1.040
4	.020	.048	.095	.214	1.012
5	.024	.042	.092	.208	0.992
6	.016	.046	.094	.211	1.006
7	.014	.049	•099	.196	0.972
Æ	.0186	.0476	.0976	.2094	1.004
SD	.0049	.0046	.0067	.0076	.0236
cv	26.2	9.8	6.9	3.6	2.4
Percent Recovery	93	95	98	105	100

STATE STITUTE SERVING FERRICH LITTLE SCHOOL SCHOOLS STATES SUBSTRACE THROUGH BUILDING 1955

FLAME Zn (mg/L)

Replicate	<u>(1)</u>	(2)	Spike (3)	<u>(4)</u>	<u>(5)</u>
		 -	<u> </u>	<u> </u>	<u> </u>
Expected	.010	.020	.100	.50	2.000
. 1	.032	.042	.102	.477	1.913
2	.034	.038	.110	.4 91	1.935
3	.030	.041	.101	.474	1.916
4	.034	.038	.111	.486	1.937
5	.030	.042	.101	.476	1.920
6	.036	.039	.111	.492	1.932
7	.032	.041	.105	.485	1.926
₹	.0327	.0401	.1059	.483	1.926
SD	.0024	.0018	.0047	.0073	.0095
cv	7.22	4.42	4.44	1.52	0.49
Percent Recovery	127	200	106	97	96

ICP Zn (mg/L)

			Spike		
Replicate	<u>(1)</u>	<u>(2)</u>	<u>(3)</u>	<u>(4)</u>	<u>(5)</u>
Expected	.020	.050	.100	.500	2.000
1	.014	.048	.089	.475	1.897
2	.018	.053	.084	.494	2.000
3	.018	.059	.089	.490	1.860
4	.023	.046	.090	.502	1.864
5	.022	.046	.097	.494	1.890
6	.026	.049	.096	.501	1.862
7	.020	.051	.103	.499	1.874
Ā	.0203	.0502	.093	.494	1.892
SD	.0039	.0046	.0064	.0093	.0495
cv	19.53	9.16	6.91	1.9	2.6
Percent Recovery	102	100	93	99	95

DL = .012

ASSAS SUCCESSON TO SERVICE PROCESSORS PROGRAMMENT

TRACE ORGANICS

Accuracy was determined as percent error on a spike added to a sample and analyzed in septuplet. Several different sample sources were utilized including tap water, EEWTP blended influent and EEWTP finished water.

Method detection limits were defined as three times the standard deviation (precision).

Method Key:

SOUTH THE WASHINGTON

ANY LEADER LEASERED INVESTOR LEAVENCE XTORING

BN = Base-neutral extractables
CLS = Closed-loop stripping
ACIDS = Acid extractables

MAC = Methylated acid extractables VOA = Volatile organics analysis

PEST/PCB = Pesticides and polychlorinated biphenyls

HERB = Herbicides

LLE = Liquid-liquid extraction (pentane)

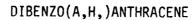
BENZO(K)FLUORANTHENE

Replicates	BN (20 ppb)
1	20
2	18
3	20
4	20
5	27
6	24
7	19
Mean	21
Standard Deviation	3.2
Percent Standard Deviation	15
Actual Value	20
Percent Error	+5
3 Times S.D.	9.6

STATE VERSION PROPER VERSION INCOME.

BENZO(G,H,I)PERYLENE

Replicates	BN (20 ppb)
1	11
2	15
3	21
4	16
5	12
6	8
7	21
Mean	15
Standard Deviation	5.0
Percent Standard Deviation	33
Actual Value	20
Percent Error	-25
3 Times S.D.	15



Replicates	BN (20 ppb)
1	21
2	15
3	21
4	18
5	22
6	15
7	20
Mean	19
Standard Deviation	2.9
Percent Standard Deviation	15
Actual Value	20
Percent Error	-5
3 Times S.D.	8.7

BENZO(A)PYRENE

Replicates	BN (20 ppb)
1	21
2	26
3	19
4	29
5	22
6	18
7	18
Mean	22
Standard Deviation	4.2
Percent Standard Deviation	19
Actual Value	20
Percent Error	+10
3 Times S.D.	13

1,1'-OXYBIS(2-CHLOROETHANE)

Replicates	BN (20 ppb)	CLS (0.1 ppb)
1 .	19	0.112
2	18	0.097
3	19	0.085
4	19	0.077
5	22	0.085
6	18	0.078
7	19	ND
Mean	19	0.076
Standard Deviation	1.4	0.036
Percent Standard Deviation	7.0	47
Actual Value	20	0.100
Percent Error	-5	-24
3 Times S.D.	4.2	0.11

SECOND MANAGE LANGE LANGE LANGE AND SECOND SECOND

BENZO(B)FLUORANTHENE

Replicates	BN (20 ppb)
1	20
2	18
3	27
4	26
5	26
6	18
7	18
Mean	22
Standard Deviation	4.3
Percent Standard Deviation	19
Actual Value	20
Percent Error	+10
3 Times S.D.	13

PYRENE

Replicates	BN (20 ppb)
1	20
2	18
3	21
4	19
5	23
6	18
7	20
Mean	20
Standard Deviation	1.8
Percent Standard Deviation	8.9
Actual Value	20
Percent Error	0
3 Times S.D.	5.4

SACRETARY CONTROL OF THE PROPERTY OF THE PROPE

CHRYSENE

Replicates	BN (20 ppb)
1	20
2	17
3	20
4	19
5	23
6	17
7	19
Mean	19
Standard Deviation	2.1
Percent Standard Deviation	11
Actual Value	20
Percent Error	-5
3 Times S.D.	6.3

BENZO(A)ANTHRACENE

Replicates	BN (20 ppb)
1	19
2	16
3	21
4	18
5	23
6	18
7	18
Mean	19
Standard Deviation	2.3
Percent Standard Deviation	12
Actual Value	20
Percent Error	-5
3 Times S.D.	6.9

N-NITROSODIPHENYLAMINE

Replicates	BN (20 ppb)
1	15
2	11
3	11
4	12
5	11
6	12
7	14
Mean	12
Standard Deviation	1.6
Percent Standard Deviation	13
Actual Value	20
Percent Error	-40
3 Times S.D.	4.8



ACENAPHTHENE

Replicates	BN (20 ppb)
1	21
2	20
3	22
4	20
5	22
6	20
7	21
Mean	21
Standard Deviation	0.90
Percent Standard Deviation	4.3
Actual Value	20
Percent Error	+5
3 Times S.D.	2.7



DIMETHYLPHTHALATE

Replicates	BN (20 ppb)
	·····
1	17
2	14
3	9
4	14
5	19
6	16
7	9
Mean	14
Standard Deviation	3.8
Percent Standard Deviation	27
Actual Value	20
Percent Error	-30
3 Times S.D.	11

ACENAPHTHYLENE

Replicates	BN (20 ppb)
1	19
2	18
3	18
4	18
5	20
6	18
7	19
Mean	19
Standard Deviation	0.79
Percent Standard Deviation	4.2
Actual Value	20
Percent Error	-5
3 Times S.D.	2.4

CONTRACTOR LANGUAGE SANGER SANGERS

ISOPHORONE

Replicates	BN (20 ppb)
1	20
2	18
3	19
4	19
5	21
6	18
7	19
Mean	19
Standard Deviation	1.1
Percent Standard Deviation	5.6
Actual Value	20
Percent Error	-5
3 Times S.D.	3.3



NITROBENZENE

Replicates	BN (20 ppb)
1	17
2	17
3	17
4	17
5	19
6	17
7	17
Mean	17
Standard Deviation	0.76
Percent Standard Deviation	4.4
Actual Value	20
Percent Error	-15
3 Times S.D.	2.3



1,1'-(METHYLENEBIS(OXY))-BIS-2-CHLOROETHANE

Replicates	BN (20 ppb)
1	24
2	24
3	26
4	24
5	26
6	24
7	25
Mean	25
Standard Deviation	0.95
Percent Standard Deviation	3.9
Actual Value	20
Percent Error	+25
3 Times S.D.	2.9

2,2'-OXYBIS(2-CHLOROPROPANE)

ALLE VOLVEYOR CHREEKEET THE REAL KNIGHT WAS

Replicates	BN (20 ppb)
1	19
2	19
3	19
4	19
5	22
6	19
7	19
Mean	19
Standard Deviation	1.1
Percent Standard Deviation	5.8
Actual Value	20
Percent Error	-5
3 Times S.D.	3.3

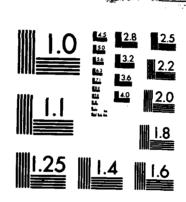
3,3'-DICHLOROBENZIDINE

Replicates	BN (20 ppb)
1	16
2	10
3	8
4	9
5	11
6	10
7	10
Mean	11 .
Standard Deviation	2.6
Percent Standard Deviation	24
Actual Value	20
Percent Error	-45
3 Times S.D.	7.8



Replicates	BN (20 ppb)
1	19
2	17
3	18
4	18
5	19
6	17
7	17
Mean	18
Standard Deviation	0.90
Percent Standard Deviation	5.0
Actual Value	20
Percent Error	-10
3 Times S.D.	2.7

OPERATION MAINTENANCE AND PERFORMANCE EVALUATION OF THE POTOMAC ESTUARY E. (U) MONTGOMERY (JAMES M) CONSULTING ENGINEERS INC PASADENA CA J M MONTGOMETRY SEP 83 MMR-83-MA-VOL-1 DACM31-88-C-0041 F/G 13/2 AD-A136 865 3/6 -UNCLÁSSIFIED NL



2000 1995 - The read I service 1858 - The service

MICROCOPY RESOLUTION TEST CHART

NATIONAL BUREAU OF STANDARDS-1963-A

- The Ithia

1,1-DICHLOROETHENE

Replicates	VOA (1 ppb)
1	0.9
2	0.9
3	1.3
4	1.1
5	0.9
6	0.9
7	1.0
Mean	1.00
Standard Deviation	0.15
Percent Standard Deviation	15
Actual Value	1.00
Percent Error	0
3 Times S.D.	0.45

TRICHLOROFLUOROMETHANE

<u>Replicates</u>	VOA (1 ppb)
1	0.8
2	0.8
3	0.9
4	0.8
5	0.6
6	1.0
7	1.0
Mean	0.84
Standard Deviation	0.14
Percent Standard Deviation	16.6
Actual Value	1.0
Percent Error	-16
3 Times S.D.	0.42

DICHLOROMETHANE

Replicates	VOA (1 ppb)
1	1.8
2	1.4
3	2.4
4	1.9
5	1.4
6	0.9
7	3.1
Mean	1.84
Standard Deviation	0.73
Percent Standard Deviation	39
Actual Value	1.43
Percent Error	+29
3 Times S.D.	2.2

CHLOROETHANE

Replicates	VOA (1 ppb)
1	0.8
2	0.9
3	0.8
4	0.9
5	0.8
6	0.8
7	1.0
Mean	0.86
Standard Deviation	0.079
Percent Standard Deviation	9.2
Actual Value	1.0
Percent Error	-14
3 Times S.D.	0.24

BROMOMETHANE

STATES STATES STATES STATES STATES STATES

CONTRACTOR TO SECURE TRACTOR TO SECURITY

Replicates	VOA (1 ppb)
1	1.0
2	1.1
3	1.0
4	0.8
5	1.0
6	1.0
7	0.8
Mean	0.96
Standard Deviation	0.11
Percent Standard Deviation	12
Actual Value	1.0
Percent Error	-4
3 Times S.D.	0.33



CHLOROMETHANE

Replicates	VOA (1 ppb)
1	1.0
2	0.8
3	0.7
4	0.7
5	1.0
6	0.9
7	0.7
Mean	0.83
Standard Deviation	0.14
Percent Standard Deviation	17
Actual Value	1
Percent Error	-17
3 Times S.D.	0.42



VINYL CHLORIDE

Replicates	VOA (1 ppb)
1	0.9
2	1.0
3	1.0
4	0.8
5	1.0
6	0.8
7	0.9
Mean	0.91
Standard Deviation	0.090
Percent Standard Deviation	9.8
Actual Value	1.0
Percent Error	-9
3 Times S.D.	0.27



BUTYLBENZYLPHTHALATE

Replicates	BN (20 ppb)
1	15
2	13
3	11
4	13
5	16
6	11
7	10
Mean	13
Standard Deviation	2.2
Percent Standard Deviation	17
Actual Value	20
Percent Error	-35
3 Times S.D.	6.6



N-NITROSODIMETHYLAMINE

Replicates	BN (20 ppb)
1	14
2	26
3	14
4	15
5	20
6	14
7	14
Mean	17
Standard Deviation	4.6
Percent Standard Deviation	28
Actual Value	20
Percent Error	-15
3 Times S.D.	14



Replicates	VOA (1 ppb)	LLE ECD (1.5 ppb)
1	1.0	1.4
2	1.0	1.4
3	1.1	1.4
4	1.1	1.4
5	1.1	1.4
6	1.9	1.4
7	1.0	1.4
Mean	1.04	1.4
Standard Deviation	0.054	0
Percent Standard Deviation	5	0
Actual Value	1.0	12.2
Percent Error	+4	+14
3 Times S.D.	0.16	0.11

LADVINIA CITATOR SAMPLES LADVINIA CARPET

BROMOCHLOROMETHANE

Replicates	VOA (1 ppb)
1	1.1
2	1.1
3	1.1
4	1.1
5	0.7
6	0.7
7	0.7
Mean	0.93
Standard Deviation	0.21
Percent Standard Deviation	2.3
Actual Value	1.00
Percent Error	- 7
3 Times S.D.	0.63

1,1,1-TRICHLOROETHANE

Replicates	VOA (1 ppb)
1	1.0
2	0.9
3	1.0
4	1.0
5	1.0
6	1.1
7	1.1
Mean	1.0
Standard Deviation	0.071
Percent Standard Deviation	6.8
Actual Value	1.0
Percent Error	0
3 Times S.D.	0.21

ACETONE

Replicates	VOA (1 ppb)
4	
1	1.2
2	1.4
3	0.9
4	1.0
5	1.0
6	0.9
7	1.0
Mean	1.06
Standard Deviation	0.18
Percent Standard Deviation	17
Actual Value	1.0
Percent Error	+6
3 Times S.D.	0.54

1,2-DIBROMO-3-CHLOROPROPANE

Replicates	VOA (1 ppb)
1	1.2
2	1.2
3	1.2
4	1.1
5	1.2
6	1.2
7	1.0
Mean	1.16
Standard Deviation	0.079
Percent Standard Deviation	6.8
Actual Value	1.0
Percent Error	+16
3 Times S.D.	0.24

BENZENE

Replicates	VOA (1 ppb)
1	1.0
2	1.0
3	1.0
4	1.0
5	1.0
6	1.0
7	1.1
Mean	1.01
Standard Deviation	0.038
Percent Standard Deviation	37
Actual Value	1.0
Percent Error	+1
3 Times S.D.	6.11

CIS-1,3-DICHLOROPROPENE

Replicates	VOA (1 ppb)
1	1.0
2	1.1
3	1.1
4	1.1
5	1.1
6	1.1
7	1.1
Mean	1.09
Standard Deviation	0.038
Percent Standard Deviation	3.5
Actual Value	1.0
Percent Error	+9
3 Times S.D.	0.11

1,2-DICHLOROETHANE

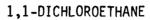
Replicates	VOA (1 ppb)
1	1.0
2	1.1
3	1.1
4	1.0
5	0.7
6	1.1
. 7	1.0
Mean	1.00
Standard Deviation	0.14
Percent Standard Deviation	14
Actual Value	1.0
Percent Error	0
3 Times S.D.	0.42

TETRAHYDROFURAN

Replicates	VOA (1 ppb)
1	1.0
2	1.2
3	1.0
4	1.0
5	1.1
6	1.1
7	1.1
Mean	1.07
Standard Deviation	0.076
Percent Standard Deviation	7.1
Actual Value	1.00
Percent Error	+7
3 Times S.D.	0.23

2-BUTANONE

Replicates	VOA (1 ppb)
1	1.2
2	0.9
3	0.6
4	1.8
5	0.8
6	0.9
7	1.0
Mean	1.03
Standard Deviation	0.39
Percent Standard Deviation	38
Actual Value	1.00
Percent Error	+3
3 Times S.D.	1.2



Replicates	VOA (1 ppb)
1	1.2
2	1.3
3	1.3
4	1.2
5	0.9
6	0.9
7	0.9
Mean	1.10
Standard Deviation	0.19
Percent Standard	17.4
Deviation	1.0
Actual Value	
Percent Error	+10
3 Times S.D.	0.57

TRANS-1,2-DICHLOROETHENE

Replicates	VOA	(1 ppb)
1		1.3
2		1.3
3		1.4
4		1.7
5		1.3
6		1.2
7		1.3
Mean		1.36
Standard Deviation		0.16
Percent Standard Deviation		12
Actual Value		1.0
Percent Error	+	36
3 Times S.D.		0.48



Replicates	VOA	(1 ppb)
1		1.1
2		1.1
3		1.1
4		1.1
5		1.0
6		1.0
7		1.1
Mean		1.07
Standard Deviation		0.049
Percent Standard Deviation		4.6
Actual Value		1.00
Percent Error		+7
3 Times S.D.		0.15

HEXACHLOROBENZENE

Replicates	CLS (0.100 ppb)	BN (20 ppb)
1	0.092	18
2	0.073	18
3	0.094	19
4	0.054	18
5	0.071	20
6	0.082	18
7	0.103	19
Mean	0.081	19
Standard Deviation	0.017	0.29
Percent Standard Deviation	21	4.2
Actual Value	0.100	20
Percent Error	-19	-5
3 Times S.D.	0.051	2.4

(UII) VSSSSSS (UNICCO SPENSSS | DZGGUR SPOSSZ S

1-CHLORO-4-PHENOXYBENZENE

Replicates	CLS (0.100 ppb)	BN (20 ppb)
1	0.114	. 24
2	0.100	22
3	0.089	25
4	0.081	23
5	0.091	22
6	0.088	19
7	0.093	18
Mean	0.094	22
Standard Deviation	0.011	2.5
Percent Standard Deviation	11	12
Actual Value	0.100	20
Percent Error	-6	+10
3 Times S.D.	0.033	7.5

1,2-DIPHENYLHYDRAZINE

Replicates	CLS (0.100 ppb)	BN (20 ppb)
1	0.060	33
2	0.051	37
3	0.100	34
4	0.050	35
5	0.087	35
6	0.044	38
7	0.128	31
Mean	0.074	35
Standard Deviation	0.032	2.4
Percent Standard Deviation	42	6.8
Actual Value	0.100	40
Percent Error	-26	-13
3 Times S.D.	0.096	2.2

FLUORENE

Replicates	CLS (0.100 ppb)	BN (20 ppb)
1	0.082	22
2	0.068	21
3	0.093	23
4	0.047	21
5	0.085	21
6	0.085	20
7	0.131	22
Mean	0.084	21
Standard Deviation	0.026	0.98
Percent Standard Deviation	30	4.6
Actual Value	0.100	20
Percent Error	-16	+5
3 Times S.D.	0.074	2.9

Innibion Careeres Labarage Labarina statemen assesses

FLUORANTHENE

Replicates	BN (10 ppb)
1	8.6
2	11.6
3	7.3
4	8.3
5	7.8
6	8.7
7	10.2
Mean	8.9
Standard Deviation	1.5
Percent Standard Deviation	17
Actual Value	10
Percent Error	11
3 Times S.D.	4.5

2-CHLORONAPHTHALENE

Replicates	CLS (0.100 ppb)	BN (20 ppb)
1	0.111	22
2	0.092	22
3	0.091	18
4	0.066	14
5	0.081	18
6	0.111	17
7	0.094	15
Mean	0.092	18
Standard Deviation	0.016	31
Percent Standard Deviation	17	17
Actual Value	0.100	20
Percent Error	-8	-10
3 Times S.D.	0.048	9.3

HEXACHLOROCYCLOPENTADIENE

Replicates	CLS (0.100 ppb)	BN (20 ppb)
1	0.052	25
2	0.195	22
3	0.339	29
4	0.039	16
5	0.039	27
6	0.039	24
7	0.130	17
Mean	0.119	23
Standard Deviation	0.114	4.9
Percent Standard Deviation	96	21
Actual Value	0.100	20
Percent Error	+19	+15
3 Times S.D.	0.342	15

HEXACHLOROBUTADIENE

Replicates	CLS (0.100 ppb)	BN (20 ppb)
1	0.101	15
2	0.109	23
3	0.102	29
4	0.059	21
5	0.077	23
6	0.096	23
7	0.089	21
Mean	0.090	22
Standard Deviation	0.017	4.1
Percent Standard Deviation	19	19
Actual Value	0.100	20
Percent Error	-10	+10
3 Times S.D.	0.051	12

THE TAXABLE CONTROL OF THE PROPERTY OF THE PROPERTY SECRETARY SECRETARY SECRETARY OF THE PROPERTY OF

CARBON TETRACHLORIDE

Replicates	VOA (1 ppb)	LLE (1 ppb)
1	1.10	1.5
2	0.94	1.5
3	1.12	1.5
4	1.01	1.4
5	1.35	1.4
6	1.24	1.5
7	1.07	1.5
Mean	1.12	1.47
Standard Deviation	0.14	0.05
Percent Standard Deviation	12	3
Actual Value	1.13	1.57
Percent Error	-1	-13
3 Times S.D.	0.42	0.15

HEXACHLOROETHANE

Replicates	CLS (0.100 ppb)	BN (20 ppb)
1	0.116	15
2	0.111	14
3	0.098	17
4	0.071	12
5	0.080	18
6	0.090	15
7	0.078	11
Mean	0.092	15
Standard Deviation	0.017	2.5
Percent Standard Deviation	19	17
Actual Value	0.100	20
Percent Error	-8	-25
3 Times S.D.	0.051	7.5

TRICHLOROETHENE

Replicates	CLS (0.100 ppb)	VOA (1.0 ppb)	LLE (2.01 ppb)
1	0.106	1.0	2.5
2	0.109	1.2	2.5
3	0.016	1.1	2.6
4	0.056	1.1	2.3
5	0.143	0.7	2.3
6	0.107	1.2	2.5
7	0.082	1.2	2.5
Mean	0.088	1.07	2.46
Standard Deviation	0.042	0.18	0.11
Percent Standard Deviation	47	17	4
Actual Value	0.100	1.0	2.36
Percent Error	-12	+7	+4
3 Times S.D.	0.126	0.54	0.33

passi issesse monthe especie especies apprint excess course assesse barren souther terms

TETRACHLOROETHENE

Replicates	CLS (0.100 ppb)	VOA (0.2 ppb)	LLE (1.49 ppb)
1	0.141	1.11	2.3
2	0.104	1.00	2.4
		1.00	
3	0.105	1.14	2.4
4	0.095	1.10	2.1
5	0.150	1.41	2.2
6	0.102	1.23	2.4
7	0.088	1.13	2.3
Mean	0.112	1.16	2.30
Standard Deviation	0.024	0.13	0.12
Percent Standard Deviation	21	11	5
Actual Value	0.100	1.45	1.71
Percent Error	+12	-20	+35
3 Times S.D.	0.072	0.39	0.36

BROMOBENZENE

Replicates	CLS (0.100 ppb)	BN (10 ppb)
1	0.110	5.8
2	0.101	8.3
3	0.101	7.3
4	0.093	6.2
5	0.094	5.7
6	0.095	9.6
7	0.085	9.7
Mean	0.097	. 7.5
Standard Deviation	0.0079	1.3
Percent Standard Deviation	8.1	17
Actual Value	0.100	10
Percent Error	-3	-25
3 Times S.D.	0.024	3.9

1-CHLORONAPHTHALENE

Replicates	CLS (0.100 ppb)	BN (10 ppb)
1	0.098	7.9
2	0.083	8.1
3	0.085	8.0
4	0.060	8.5
5	0.075	7.5
6	0.091	9.2
7	0.109	8.6
Mean	0.086	8.3
Standard Deviation	0.016	0.6
Percent Standard Deviation	18	7
Actual Value	0.100	10
Percent Error	-14	-17
3 Times S.D.	0.048	1.8

1-BROMO-4-PHENOXYBENZENE

<u>Replicates</u>	CLS (0.200 ppb)	BN (11 ppb)
	0.200 pps/	bit (11 ppd)
1	0.115	7.7
2	0.100	7.6
3	0.084	9.6
4	0.088	12
5	0.094	8.3
6	0.088	7.9
7	0.092	9.0
Mean	0.094	8.9
Standard Deviation	0.010	1.6
Percent Standard Deviation	11	18
Actual Value	0.100	11
Percent Error	-6	-19
3 Times S.D.	0.030	4.8

poce material restrict communication beautiful common

4-CHLORO-3-METHYLPHENOL

Replicates	MAC (20 ppb)	ACIDS (10.5 ppb)
1	16	6.5
2	17	9.0
3	20	8.0
4	19	4.5
5	13	5.7
6	18	9.2
7		6.9
Mean	17.2	7.1
Standard Deviation	2.5	1.7
Percent Standard Deviation	14	24
Actual Value	20	10.5
Percent Error	-14	-32
3 Times S.D.	7.5	5.1

2,3,5-TRICHLOROPHENOL

Replicates	MAC (20 ppb)	ACIDS (10 ppb)
1	15	4.2
2	16	9.5
3	19	9.4
4	19	5.6
5	13	5.7
6	17	10.0
7		10.0
Mean	16.5	7.8
Standard Deviation	2.4	2.5
Percent Standard Deviation	14	32
Actual Value	20	9
Percent Error	-17	-13
3 Times S.D.	7.2	7.5

2,4,5-TRICHLOROPHENOL

Replicates	MAC (20 ppb)	ACIDS (9.5 ppb)
1	17	9.0
2	21	10.1
3	17	7.8
4	18	6.6
5	13	6.0
6	20	10.7
7		6.2
Mean	17.6	8.1
Standard Deviation	2.8	1.9
Percent Standard Deviation	16	24
Actual Value	20	9.5
Percent Error	-12	-15
3 Times S.D.	8.4	5.7

2,4,6-TRICHLOROPHENOL

Replicates	MAC (20 ppb)	ACIDS (10.5 ppb)
1	15	14
2	16	11
3	19	7.0
4	17	7.6
5	13	9.5
6	18	8.8
7		8.3
Mean	16.3	9.5
Standard Deviation	2.2	2.4
Percent Standard Deviation	13	26
Actual Value	20	10.5
Percent Error	-18	-10
3 Times S.D.	6.6	7.2

2,3,6-TRICHLOROPHENOL

Replicates	MAC (20 ppb)	ACIDS (9ppb)
1	14	6.9
2	15	11
3	20	11.3
4	17	5.3
5	12	7.7
6	17	10.7
7		7.5
Mean	15.8	8.6
Standard Deviation	2.8	2.3
Percent Standard Deviation	18	26
Actual Value	20	9
Percent Error	-21	-4
3 Times S.D.	8.4	6.9

2-CHLOROPHENOL

Replicates	MAC (20 ppb)	ACIDS (8.8 ppb)
1	19	9.2
2	19	9.6
3	24	7.6
4	19	5.4
5	16	6.8
6	22	8.4
7		8.5
Mean	19.8	7.9
Standard Deviation	2.8	1.5
Percent Standard Deviation	14	18
Actual Value	20	8.8
Percent Error	-1	-10
3 Times S.D.	8.4	4.5

2,4-DICHLOROPHENOL

Replicates	MAC (20 ppb)	ACIDS (10.5 ppb)
1	14	10
2	16	13
3	18	11
4	17	6.9
5	12	9.5
6	18	12
;		9.0
Mean	15.8	10
Standard Deviation	2.4	2
Percent Standard Deviation	15	20
Actual Value	20	10.5
Percent Error	-21	-5
3 Times S.D.	7.2	6

Secretary description of the secretary property and the secretary description of the secretary s

PHENOL

Replicates	MAC (20 ppb)	ACIDS (9.5 ppb)
1	15	9.8
2	17	11
3	21	11
4	17	7.1
5	13	6.8
6	17	8.6
7		9.1
Mean	16.7	9.1
Standard Deviation	2.7	1.7
Percent Standard Deviation	16	19
Actual Value	20	9.5
Percent Error	-16	-4
3 Times S.D.	8.1	5.1

PENTACHLOROPHENOL

<u>Replicates</u>	MAC (20 ppb)	ACIDS (42 ppb)
1	10	47
2	11	34
3	12	34
4	11	26
5	8	34
6	11	24
7		16
Mean	10.5	31
Standard Deviation	1.4	9.8
Percent Standard Deviation	13	32
Actual Value	20	42
Percent Error	-47	-26
3 Times S.D.	4.2	29

PHENANTHRENE

Replicates	CLS (0.100 ppb)	BN (20 ppb)
1	0.033	18
2	0.042	17
3	0.091	20
4	0.060	17
5	0.113	21
6	0.029	16
7	ND	18
Mean	0.053	18
Standard Deviation	0.039	1.8
Percent Standard Deviation	74	9.8
Actual Value	0.100	20
Percent Error	-47	-10
3 Times S.D.	0.117	5.4

ANTHRACENE

Replicates	CLS (0.100 ppb)	BN (20 ppb)
1	0.025	20
2	0.039	23
3	0.080	22
4	0.073	24
5	0.067	20
6	0.075	23
7	ND	19
Mean	0.051	22
Standard Deviation	0.030	1.9
Percent Standard	59	8.8
Deviation	0.100	20
Actual Value		
Percent Error	-49	+10
3 Times S.D.	0.090	5.7

consisted endicate consisted limitalists absorbed

1,2-DICHLOROBENZENE

Replicates	CLS (0.100 ppb)	VOA (1.0 ppb)	BN (20 ppb)
1	0.106	1.1	18
2	0.101	1.2	19
3	0.098	1.0	21
4	0.088	1.1	19
5	0.092	1.1	20
6	0.094	1.1	18
7	0.091	1.0	18
Mean	0.095	1.09	19
Standard Deviation	0.0063	0.069	1.2
Percent Standard Deviation	6.6	6.4	6.1
Actual Value	0.100	1.0	20
Percent Error	-5	+9	-5
3 Times S.D.	0.019	0.21	3.6

1,2-DIBROMOETHANE

Replicates	CLS (0.100 ppb)	VOA (1.0 ppb)
1	0.084	1.1
2	0.065	1.1
3	0.067	1.1
4	0.092	1.1
5	0.105	1.1
6	0.081	1.1
7	0.054	1.1
	0.070	1 1
Mean	0.078	1.1
Standard Deviation	0.018	0
Percent Standard Deviation	22	0
Actual Value	0.100	1.0
Percent Error	-22	+10
3 Times S.D.	0.054	< 0.11

1,1,2-TRICHLOROETHANE

Replicates	CLS (0.100 ppb)	VOA (1.0 ppb)
1	0.081	1.0
2	0.061	1.1
3	0.034	1.1
4	0.089	1.1
5	0.104	1.1
6	0.082	1.1
7	0.053	1.1
Mean	0.072	1.09
Standard Deviation	0.024	0.038
Percent Standard Deviation	33	3.5
Actual Value	0.100	1.0
Percent Error	-28	+9
3 Times S.D.	0.072	0.11

1,3-XYLENE/1,4-XYLENE

<u>Replicates</u>	CLS (0.100 ppb)	VOA (2.0 ppb)
1	0.129	2.0
2	0.099	1.9
3	0.104	1.9
4	0.095	2.3
5	0.119	2.1
6	0.093	2.0
7	0.094	2.0
Mean	0.105	2.03
Standard Deviation	0.014	0.14
Percent Standard Deviation	13	6.8
Actual Value	0.100	2.00
Percent Error	+5	+1
3 Times S.D.	0.042	0.42

BROMOFORM

Replicates	GLS (0.100 ppb)	VOA (1.0 ppb)	LLE ECD (1 ppb
1	0.097	1.0	2.1
2	0.073	1.4	2.0
3	0.071	1.1	2.1
4	0.086	1.2	2.1
5	0.086	1.4	2.0
6	0.079	1.4	2.2
7	0.058	1.5	2.1
Mean	0.079	1.29	2.07
Standard Deviation	0.013	0.19	0.08
Percent Standard Deviation	16	15	4
Actual Value	0.100	1.57	1.60
Percent Error	-21	-18	+29
3 Times S.D.	0.039	0.57	0.24

BROMODICHLOROMETHANE

Replicates	CLS (0.100 ppb)	VOA (1.0 ppb)	LLE (1.4)
1	0.065	1.0	2.3
2 ,	0.055	1.1	2.3
3	0.021	1.1	2.3
4	0.067	1.1	2.2
5	0.100	1.0	2.1
6	0.067	1.1	2.3
7	0.048	1.1	2.4
Mean	0.060	1.07	2.27
Standard Deviati	on 0.024	0.049	0.10
Percent Standard Deviation	40	4.6	4
Actual Value	0.100	1.1	1.82
Percent Error	-40	+7	+25
3 Times S.D.	0.027	0.15	0.30
Mean Standard Deviati Percent Standard Deviation Actual Value Percent Error 3 Times S.D.			
	A-2-131		

TOLUENE

Replicates	CLS (0.100 ppb)	VOA (1 ppb)
1	0.155	1.0
2	0.103	1.1
3	0.089	1.0
4	0.096	1.0
5	0.164	1.0
6	-0.102	1.0
7	0.101	1.0
Mean	0.116	1.01
Standard Deviation	0.030	0.038
Percent Standard Deviation	26	3.7
Actual Value	0.100	1.0
Percent Error	+16	+1
3 Times S.D.	0.090	0.11

DIBROMOCHLOROMETHANE

Replicates	CLS (0.100 ppb)	VOA (1.0 ppb)	LLE (1.78 ppb)
1	0.083	1.0	2.1
2	0.068	1.1	2.0
3	0.068	1.1	2.1
4	0.094	1.1	2.0
5	0.101	1.4	1.9
6	0.080	1.1	2.1
7	0.056	1.1	2.1
Mean	0.079	1.13	2.04
Standard Deviation	0.016	0.13	0.08
Percent Standard Deviation	20	11	4
Actual Value	0.100	1.0	1.91
Percent Error	-21	+13	7
3 Times S.D.	0.048	0.39	0.24

CHLOROBENZENE

Replicates	CLS (0.100 ppb)	VOA (1.0 ppb)
1	0.108	1.0
2	0.104	1.0
3	0.098	1.0
4	0.097	1.1
5	0.097	1.1
6	0.094	1.1
7	0.087	1.1
Mean	0.098	1.06
Standard Deviation	0.0068	0.054
Percent Standard Deviation	6.9	5.1
Actual Value	0.100	1.0
Percent Error	-2	+6
3 Times S.D.	0.020	0.16

ETHYLBENZENE

SACRET STREET STREETS RESIDENCE STREETS ASSESSED

SCHOOL MANAGER HANGARM CONTRACTOR

Replicates	CLS (0.100 ppb)	VOA (1.0 ppb)
1	0.126	1.0
2	0.101	1.0
3	0.102	1.0
4	0.096	1.1
5	0.114	1.0
6	0.093	1.0
7	0.092	1.0
Mean	0.103	1.01
Standard Deviation	0.012	0.038
Percent Standard Deviation	12	3.7
Actual Value	0.100	1.0
Percent Error	+3	+1
3 Times S.D.	0.036	0.11

1,2-DICHLOROPROPANE

<u>Replicates</u>	CLS (0.100 ppb)	VOA (1.0 ppb)
1	0.066	1.1
2	0.061	1.1
3	0.0098	1.1
4	0.055	1.0
5	0.097	1.0
6	0.087	1.0
7	0.055	1.1
Mean	0.062	1.06
Standard Deviation	0.028	0.054
Percent Standard Deviation	45	1.1
Actual Value	0.100	1.0
Percent Error	-38	+6
3 Times : 7.	0.084	0.16

1,2-XYLENE

Replicates	CLS (0.100 ppb)	VOA (1.0 ppb)
1	0.114	1.0
2	0.099	1.0
3	0.104	1.0
4	0.095	1.1
5	0.112	1.0
6	0.094	1.0
7	0.093	1.0
Mean	0.102	1.01
Standard Deviation	0.0087	0.038
Percent Standard Deviation	8.5	3.7
Actual Value	0.100	1.0
Percent Error	+2	+1
3 Times S.D.	0.026	0.11

1,1,2,2-TETRACHLOROETHANE

Replicates	CLS (0.100 ppb)	VOA (1.0 ppb)
1	0.087	1.0
2	0.077	0.9
3	0.057	1.1
4	0.068	1.1
5	0.061	1.0
6	0.052	1.0
7	0.042	1.0
Mean	0.063	1.01
Standard Deviation	0.015	0.069
Percent Standard Deviation	24	6.8
Actual Value	0.100	1.0
Percent Error	-37	+1
3 Times S.D.	0.045	0.21

PROPYLBENZENE

Replicates	CLS (0.100 ppb)	VOA (1.0 ppb)
1	0.097	1.1
2	0.104	0.9
3	0.106	0.9
4	0.094	1.1
5	0.103	1.0
6	0.097	1.0
7	0.095	1.0
Mean	0.099	1.0
Standard Deviation	0.0048	0.082
Percent Standard Deviation	4.8	8.2
Actual Value	0.100	1.0
Percent Error	-1	0
3 Times S.D.	0.014	0.24

1-CHLORO-4-METHYLBENZENE

Replicates	CLS (0.100 ppb)	VOA (1.0 ppb)
1	0.101	1.1
2	0.102	1.0
3	0.100	1.0
4	0.092	1.1
5	0.093	1.1
6	0.092	1.0
7	0.089	1.0
Mean .	0.096	1.04
Standard Deviation	0.0053	0.054
Percent Standard Deviation	55	5.1
Actual Value	0.100	1.0
Percent Error	-4	+4
3 Times S.D.	0.016	0.16

1,3-DICHLOROBENZENE

Replicates	CLS (0.100 ppb)	VOA (1.0 ppb)	BN (20 ppb)
1	0.110	1.1	18
2	0.100	1.0	17
3	0.103	1.0	19
4	0.091	1.1	17
5	0.093	1.1	19
6	0.092	1.1	16
7	0.090	1.0	16
Mean	0.097	1.06	17
Standard Deviation	0.0075	0.054	1.3
Percent Standard Deviation	7.8	5.1	7.3
Actual Value	0.100	1.0	20
Percent Error	-3	+6	-15
3 Times S.D.	0.023	0.16	3.9

NAPHTHALENE

Replicates	CLS (0.100 ppb)	VOA (1.0 ppb)	BN (20 ppb)
1	0.102	1.3	19
2	0.091	1.1	19
3	0.095	1.1	21
4	0.064	1.1	19
5	0.079	1.4	19
6	0.095	1.5	19
7	0.104	1.1	19
Mean	0.090	1.23	19
Standard Deviation	0.014	0.17	0.76
Percent Standard Deviation	16	14	3.9
Actual Value	0.100	1.0	20
Percent Error	-10	+23	-5
3 Times S.D.	0.042	0.51	2.3

1,4-DICHLOROBENZENE

Replicates	CLS (0.100 ppb)	VOA (1.0 ppb)	BN (20 ppb)
1	0.108	1.1	19
2	0.101	1.0	17
3	0.097	1.0	20
4	0.087	1.0	17
5	0.090	1.1	18
6	0.092	1.1	15
7	0.091	1.0	15
Mean	0.095	1.04	17
Standard Deviation	0.0073	0.054	1.9
Percent Standard Deviation	7.7	5.1	11
Actual Value	0.100	1.0	20
Percent Error	- 5	+4	-15
3 Times S.D.	0.022	0.16	5.7

ALDRIN

Replicates	PEST/PCB (1 ppb)
1	1.0
2	1.0
3	0.97
4	1.0
5	1.0
6	1.0
7	0.88
Mean	0.98
Standard Deviation	0.045
Percent Standard Deviation	5
Actual Value	1.0
Percent Error	2
3 Times S.D.	0.14



Replicates	HERBICIDE (1 ppb)
1	0.66
2	0.75
3	0.71
4	0.76
5	0.76
6	0.53
7	0.53
Mean	0.67
Standard Deviation	0.10
Percent Standard Deviation	15
Actual Value	1.0
Percent Error	33
3 Times S.D.	0.30

THE TRANSPORT OF THE PROPERTY OF THE PARTY O

2,4,5-TP

Replicates	HERBICIDES (1 ppb)
1	0.86
2	0.95
3	0.92
4	0.92
5	0.90
6	0.57
7	0.57
Mean	0.81
Standard Deviation	0.17
Percent Standard Deviation	21
Actual Value	1.0
Percent Error	19
3 Times S.D.	0.54



Replicates	PEST/PCB (1 ppb)
	•
1	0.81
2	0.82
3	0.81
4	0.89
5	0.90
6	0.85
7	0.87
Mean	0.85
Standard Deviation	-0.0399
Percent Standard Deviation	5
Actual Value	1.0
Percent Error	15
3 Times S.D.	0.12

KINN HELLEND HELLEND BESTERE FRANCISCO STEELEND

KEPONE

Replicates	PEST/PCB (1 ppb)
1	0.57
2	059
3	0.51
4	0.62
5	0.71
6	0.75
7	0.70
Mean	0.64
Standard Deviation	0.0868
Percent Standard Deviation	14
Actual Value	1.0
Percent Error	36
3 Times S.D.	0.26

ALPHA-BHC

Replicates	PEST/PCB (1 ppb)
1	1.2
2	1.2
3	1.1
4	1.2
5	1.2
6	1.2
7	1.0
Mean	1.16
Standard Deviation	0.079
Percent Standard Deviation	7
Actual Value	1.0
Percent Error	16
3 Times S.D.	0.24

Same of the first property teathers are seen to the first property that the fi

BETA-BHC

Replicates	PEST/PCB (1 ppb)
1	1.1
2	1.1
3	1.1
4	1.1
5	1.2
6	1.0
7	1.0
Mean	1.09
Standard Deviation	0.069
Percent Standard Deviation	6
Actual Value	1.0
Percent Error	9
3 Times S.D.	0.21

HEPTACHLOR

Replicates	PEST/PCB (1 ppb)
1	1.1
2	1.0
3	1.0
4	1.1
. . . .	1.1
6	1.1
7	0.94
Mean	1.0-5
Standard Deviation	0.067
Percent Standard Deviation	6
Actual Value	1.0
Percent Error	5
3 Times S.D.	0.20

HEPTACHLOR EPOXIDE

Replicates	PEST/PCB (1 ppb)
1	0.94
2	0.95
3	0.90
4	0.96
5	0.96
6	0.92
7	0.85
Mean	0.93
Standard Deviation	0.040
Percent Standard Deviation	4
Actual Value	1.0
Percent Error	7
3 Times S.D.	0.12

P,P'DDE

Replicates	PEST/PCB (1 ppb)
1	0.05
2	0.95
	0.95
3	0.92
4	0.97
5	0.98
6	0.91
7	0.89
Mean	0.94
Standard Deviation	0.033
Percent Standard Deviation	4
Actual Value	1.0
Percent Error	6
3 Times S.D.	0.099

SOLD WITHOUT MARKEY WARREN IN SELECTION

METHOXYCHLOR

Replicates	PEST/PCB (1 ppb)
1	0.85
2	0.84
3	0.83
4	0.92
5	0.86
6	0.83
7	0.87
Mean .	0.86
Standard Deviation	0.031
Percent Standard Deviation	4
Actual Value	1.0
Percent Error	14
3 Times S.D.	0.093

ENDRIN

Replicates	PEST/PCB (1 ppb)
1	0.91
2	0.91
3	088
4	0.93
5	0.94
6	0.88
. 7	0.89
Mean	0.91
Standard Deviation	0.0237
Percent Standard Deviation	3
Actual Value	1.0
Percent Error	9
3 Times S.D.	0.071

DIELDRIN

Replicates	PEST/PCB (1 ppb)
1	0.98
2	0.97
3	0.93
4	0.99
. 5	1.0
6	0.93
7	0.87
Mean	0.95
Standard Deviation	0.0459
Percent Standard Deviation	5
Actual Value	1.0
Percent Error	5
3 Times S.D.	0.14

THE STATE OF STATE OF THE PROPERTY STATES OF THE STATES.



Replicates	HERBICIDES (1 ppb)
1	0.63
2	0.47
3	0.37
4	0.72
5	0.35
6	0.19
7	0.18
Mean	0.42
Standard Deviation	0.21
Percent Standard Deviation	50
Actual Value	1.0
Percent Error	58
3 Times S.D.	0.63

P,P'DDT

Replicates	PEST/PCB (1 ppb)
1	0.83
2	0.84
3	0.84
4	0.91
5	0.87
6	0.82
7	0.86
Mean	0.85
Standard Deviation	0.0304
Percent Standard Deviation	. 4
Actual Value	1.0
Percent Error	15
3 Times S.D.	0.091

DICHLOROIODOMETHANE

Replicates	LLE (1.75 ppb)
1	1.8
2 .	2.2
3	2.1
4	1.9
5	2.1
6	2.0
7	1.7
Mean	1.97
Standard Deviation	0.18
Percent Standard Deviation	9
Actual Value	1.75
Percent Error	13
3 Times S.D.	0.54

INDENO(1,2,3-c,d)PYRENE

Replicates	BN (28 ppb)
1	28
2	46
3	41
4	24
5	24
6	41
7	40
Mean	35
Standard Deviation	9.2
Percent Standard Deviation	2.6
Actual Value	28
Percent Error	25
3 Times S.D.	28

^{*} Concentration in micrograms/liter

DIETHYLPHTHALATE

Replicates	BN (11 ppb)
1	7.4
2	10.6
3	10.5
4	12.7
5	3.6
6	9.0
7	7.4
Mean	8.7
Standard Deviation	3.0
Percent Standard Deviation	34
Actual Value	11
Percent Error	21
3 Times S.D.	9.0

DI-N-BUTYLPHTHALATE

Replicates	BN (12 ppb)
1	10.5
2	11.6
3	12.5
4	11.5
5	12.3
6	5.6
7	5.4
Mean	9.9
Standard Deviation	3
Percent Standard Deviation	31
Actual Value	12
Percent Error	18
3 Times S.D.	9.0

ATRAZINE

Replicates	PEST/PCB (20 ppb)
1	18
2	14
3	15
4	20
5	14
6	12
7	13
Mean	15
Standard Deviation	2.9
Percent Standard Deviation	19
Actual Value	20
Percent Error	25
3 Times S.D.	8.7

BIS(2-ETHYLHEXYL)PHTHALATE

Replicates	BN (20 ppb)
1	20
2	19
3	13
4	15
5	16
6	13
7	14
Mean	16
Standard Deviation	2.8
Percent Standard Deviation	18
Actual Value	20
Percent Error	20
3 Times S.D.	8.4

1-METHYL-2,6-DINITROBENZENE

Replicates	BN (22 ppb)
1	14
2	13
3	22
4	17
5	15
6	18
7	13
Mean	16
Standard Deviation	3.3
Percent Standard Deviation	20
Actual Value	22
Percent Error	27
3 Times S.D.	9.9

STATES AND A STATE STATES STATES AND STATES WHITE THE STATES STAT

DIOCTYLPHTHALATE

Replicates	BN (10.5 ppb)
1	11
2	13
3	11
4	10.4
5	17
6	9.6
7	14
Mean	12
Standard Deviation	2.6
Percent Standard Deviation	21
Actual Value	10.5
Percent Error	14
3 Times S.D.	7.8

3-CHLOROPHENOL

Replicates	ACID (10.5 ppb)
1	9.2
2	11.1
3	9.8
4	7.5
5	7.6
6	9.5
7	8.1
Mean	9.0
Standard Deviation	1.3
Percent Standard Deviation	15
Actual Value	10.5
Percent Error	14
3 Times S.D.	3.9

AROCLOR 1242

Replicates	PEST/PCB (1.22 ppb)
1	1.47
2	1.55
3	1.25
4	1.40
5	1.32
6	1.55
7	1.20
Mean	1.39
Standard Deviation	0.14
Percent Standard Deviation	10.1
Actual Value	1.22
Percent Error	+14
3 Times S.D.	0.42



Replicates	MAC (20 ppb)
1	13
2	16
3	20
4	17
5	11
6	16
7	
Mean	15.5
Standard Deviation	3.2
Percent Standard Deviation	20
Actual Value	20
Percent Error	-22
3 Times S.D.	9.6

4-CHLOROPHENOL

Replicates	MAC	(20 ppb)
1		17
2		19
3		23
4		20
5		15
6		21
7		
Mean		19.2
Standard Deviation		2.9
Percent Standard Deviation		15
Actual Value		20
Percent Error		-4
3 Times S.D.		8.7

4-NITROPHENOL

Replicates	MAC (20 ppb)
1	12
2	13
3	17
4	13
5	9
6	13
7	
Mean	12.8
Standard Deviation	2.6
Percent Standard Deviation	20
Actual Value	20
Percent Error	-36
3 Times S.D.	7.8

ETHENYLBENZENE

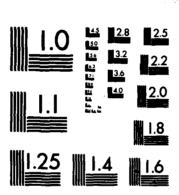
Replicates	CLS (0.100 ppb)
•	0 111
1	0.111
2	0.104
3	0.104
4	0.094
5	0.100
6	0.096
7	0.096
Mean	0.101
Standard Deviation	0.006
Percent Standard Deviation	6.0
Actual Value	0.100
Percent Error	+1
3 Times S.D.	0.018

水等原本 人名英格兰人 人名西班牙 人名西班牙

METHYLISOBORNEOL

Replicates	CLS (0.100 ppb)
1	0.115
2	0.103
3	0.080
4	0.084
5	0.077
6	0.088
7	0.088
Mean	0.091
Standard Deviation	0.014
Percent Standard Deviation	15
Actual Value	0.100
Percent Error	-19
3 Times S.D.	0.042

OPERATION MAINTENANCE AND PERFORMANCE EVALUATION OF THE POTOMAC ESTUARY E. (U) MONTGOMERY (JAMES M) CONSULTING ENGINEERS INC PASADENA CA J MONTGOMETRY SEP 83 MMA-83-MA-VOL-1 DACK31-80-C-0041 F/G 13/2 AD-A136 865 4/6 UNCLÁSSIFIED NL



MICROCOPY RESOLUTION TEST CHART

NATIONAL BUREAU OF STANDARDS-1963-A

GEOSMIN

Replicates	CLS (0.100 ppb)
1	0.116
0	0.105
2	0.105
3	0.098
4	0.085
5	0.074
6	0.085
7	0.089
Mean	0.094
Standard Deviation	0.015
Percent Standard Deviation	16
Actual Value	0.100
Percent Error	-6
3 Times S.D.	0.045

GAMMA-BHC(LINDANE)

Replicates	PEST/PCB (0.172 ppb)
1	0.179
2	0.183
3	0.184
4	0.175
5	0.178
6	0.189
7	0.170
Mean	0.180
Standard Deviation	0.0063
Percent Standard Deviation	3.5
Actual Value	0.172
Percent Error	4.6
3 Times S.D.	0.019

DELTA-BHC

SANSON STREET, STREET, SECRECA SECOND SECOND

Replicates	PEST/PCB (0.160 ppb)
1	0.165
2	0.173
3	0.173
4	0.166
5	0.168
6	0.186
7	0.161
Mean	0.170
Standard Deviation	0.0082
Percent Standard Deviation	4.8
Actual Value	0.160
Percent Error	6.2
3 Times S.D.	0.025

ENDOSULFAN I

Replicates	PEST/PCB (0.165 ppb)
1	0.185
2	0.177
3	0.175
4	0.183
5	0.171
6	0.201
7	0.174
Mean	0.181
Standard Deviation	0.010
Percent Standard Deviation	5.6
Actual Value	0.165
Percent Error	9.7
3 Times S.D.	0.03

- The contest of the second motion which the second second second which I

ENDOSULFAN II

Replicates	PEST/PCB (0.168 ppb)
1	0.188
2	0.178
3	0.176
4	0.186
5	0.176
. 6	0.207
7	0.176
Mean	0.184
Standard Deviation	0.011
Percent Standard Deviation	6.2
Actual Value	0.168
Percent Error	9.5
3 Times S.D.	0.033



Replicates	PEST/PCB (0.168 ppb)
1	0.177
2	0.187
3	0.185
4	0.177
5	0.185
6	0.200
7	0.182
Mean	0.185
Standard Deviation	0.0078
Percent Standard Deviation	4.2
Actual Value	0.168
Percent Error	10
3 Times S.D.	0.023

1,3,5 TRICHLOROBENZENE

Replicates	CLS (0.100 ppb)	VOA (1 ppb)
1	0.101	1.3
2	0.100	1.0
3	0.097	0.9
4	0.082	1.1
5	0.087	1.2
6	0.092	1.3
7	0.091	1.0
Mean	0.093	1.11
Standard Deviation	0.007	0.16
Percent Standard Deviation	7.5	14
Actual Value	0.100	1.0
Percent Error	-7	+11
3 Times S.D.	0.021	0.48



Replicates	CLS (0.100 ppb)	VOA (1.0 ppb)	BN (20 ppb)
1	0.104	1.4	18
2	0.096	1.1	18
3	0.096	1.0	22
4	0.081	1.2	15
5	0.084	1.3	21
6	0.090	1.4	18
7	0.091	1.0	16
Mean	0.092	1.20	18
Standard Deviation	0.0078	0.17	2.5
Percent Standard Deviation	8.5	14	14
Actual Value	0.100	1.0	20
Percent Error	-8	+20	-10
3 Times S.D.	0.023	0.51	7.5

THE TAXABLE COLORS LANDS LANDS WHEN WHEN THE WAS THE PASSES WHEN IN

1,2,3-TRICHLOROBENZENE

Replicates	CLS (0.100 ppb)	VOA (1 ppb)
1	0.104	1.1
2	0.097	1.1
3	0.091	1.1
4	0.079	1.1
5	0.082	1.1
6	0.091	0.9
7	0.093	1.1
Mean	0.091	1.07
Standard Deviation	0.0085	0.076
Percent Standard Deviation	9.3	7.1
Actual Value	0.100	1.0
Percent Error	-9	+7
3 Times S.D.	0.026	0.23



a series and a series of the s

SECTION 3

QUALITY CONTROL/QUALITY ASSURANCE

MONTGOMERY LABORATORIES - QUALITY ASSURANCE MANUAL

I. INTRODUCTION

The laboratory staff must have confidence in the analytical values reported to clients. This document outlines the quality assurance program that gives credibility to the values reported by this laboratory.

There are three important components in providing high quality analytical services:

- Accuracy
- Precision
- Timeliness

Analyses for inorganic, organic, and microbial consitituents of water and wastewater make up the bulk of the laboratory work load. The lab also examines other matrices and samples for specification compliance.

II. OUALITY ASSURANCE ORGANIZATION

A. Quality Assurance Manager

This person is responsible for the following:

1. Ensure that all personnel working in the laboratory follow established standard operational and safety procedures.

THE STATE OF THE PARTY OF THE P

- 2. Check procedures and ensure good housekeeping practices.
- 3. Acquaint new personnel with the rules and regulations of the laboratory.
- 4. Prepare, schedule, assign, and check quality assurance samples. Randomly introduce blind control samples without notice to the analysts.
- 5. Insure that samples are properly taken, shipped, preserved, stored and analyzed.
- 6. Insure that sample log-in and traceability are done correctly.

- 7. Preside over the Council for Quality.
- 8. Maintain the quality assurance policy.
- 9. Report directly to the Laboratory Director.

B. Council for Quality

The Council is responsible for the following:

- 1. Assist the Quality Assurance Manager in quality policy formulation and implementation.
- 2. Represent the analytical groups to the council.
- 3. Provide a forum on quality control.
- 4. Establish quality standards the group must abide by.

C. Laboratory Analytical Groups

The analytical groups are responsible for the following:

- 1. Document the quality programs.
- 2. Assign responsibility within each group to manage QC tasks.
- 3. Select members for the Quality Council.
- 4. Comply with the Quality Assurance Manual.

III. EQUIPMENT

A. Inventory

An equipment inventory is kept and maintained by the QA Manager. New equipment over \$300.00 should be entered in the inventory when it arrives. All flyers, warranties, and maintenance instructions or agreements are to be in our files.

B. Repair and Service

Service agreements have been established for the following:

- 1. Atomic Absorption Spectrophotometer and ICP: Perkin Elmer
- 2. Electron Microscope: Zeiss
- 3. Computer: Hewlett Packard
- 4. GC/MS: Finnigan

- 5. Balances: Weigh Tech
- 6. GC and HPLC: Varian

IV. SAMPLE CONTROL AND REPORTING

A. Sample Collection, Preservation and Transportation

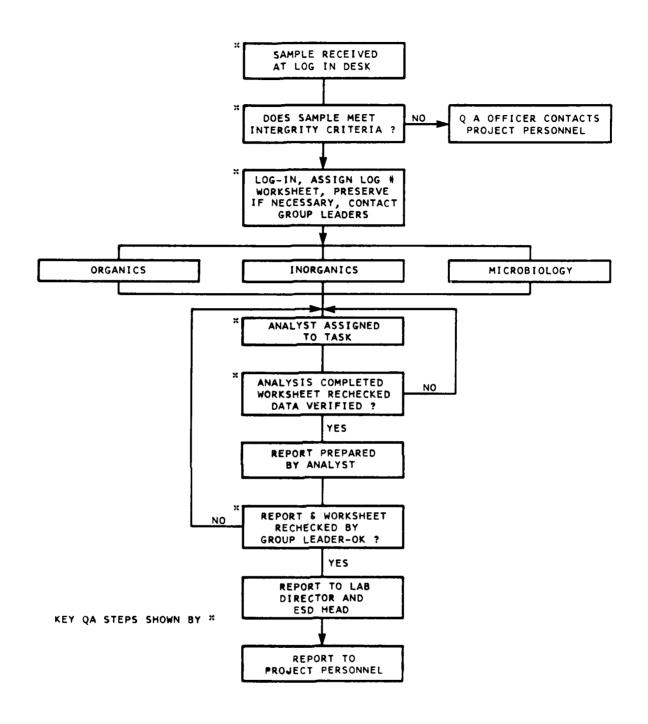
One of the key elements in Montgomery Laboratories' quality assurance program is proper preservation, shipment, and storage of samples. The laboratory staff provide guidance for selection of sampling sites and collection methods to ensure that the collected sample is representative of the water of interest. Where possible, the laboratory staff collect the samples themselves. If this is not feasible, the laboratory staff brief the clients by telephone and/or in writing on the proper methods of sample collection, preservation, storage, and shipment. Montgomery Laboratories follows the methods proposed in the Federal Register, Volume 44, No. 244, December 18, 1979 for sample collection, preservation, storage and shipment (Attachment 1).

B. Sample Receipt

The flow diagram (Figure 1) on the next page demonstrates the role of management in both the sample and QA programs. Upon arrival in Pasadena the sample control clerk inspects each shipment for broken or leaking containers and notifies the section head to decide if resampling or other appropriate action is required. Samples are then logged into the laboratory computer. The information logged in includes client name, job or purchase order number, person requesting the analysis, when samples were collected, who collected the samples, and parameters to be tested. After being logged in, the samples are stored using the guidelines in the Federal Register, Volume 44, No. 244, December 18, 1979.

C. Data Validation

Following completion of the analyses, results are validated by examining quality control parameters and any additional checks where available. For example, with respect to general minerals, after the mg/l values are entered for general minerals, a computer program calculates meq/L for the cations and anions, as well as the percent difference. This must balance within 2 percent for potable water. If not, the analyst alerts the section head, the sample is reevaluated and the QA charts are examined. When the laboratory report passes the various data validation steps the analytical report is printed. The reports are checked and signed by the analyst, reviewed and signed by the Section Head, and then forwarded to the the clerk for final distribution to the client. The reports are also forwarded to the lab director for general quality control purposes.



SAMPLE PROCESSING AT OFF-SITE LABORATORY FIGURE 1

D. Sample Handling (SIMS)

All samples received at the off-site laboratory are logged into the Sample Information Management System (SIMS) (Figure 2). This computer program keeps track of samples from the time they arrive in the laboratory until they are transmitted to the client. SIMS provides three functions:

- 1. Facilitates ready access to information on sample status (eg., which tests are completed.)
- 2. Allows analysts to schedule analyses efficiently by sorting all samples requiring a particular test.
- 3. Facilitates data manipulation in quality assurance procedures and data management.

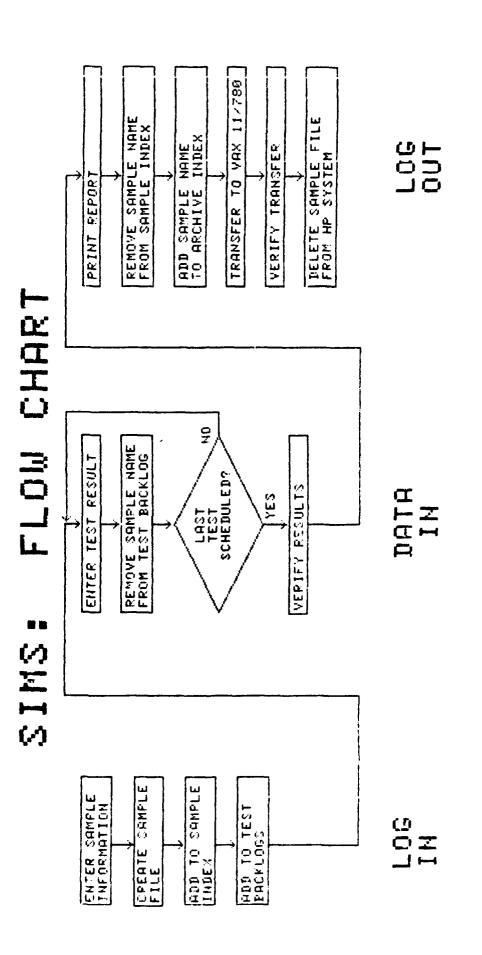
Upon arrival, each sample is assigned a laboratory I.D. number and a test number for each analysis requested or a profile which corresponds to a routine group of tests such as a general mineral analysis. In addition, a verification test is assigned for each sample which requires an employee number to be entered after a review of the data submitted for each sample. This ensures the validity of the data before a report is transmitted to the client.

Individual analysts determine what samples require a particular test by logging into SIMS and requesting those sample I.D. numbers which are incomplete and require particular tests that is still incomplete.

Senior staff members use SIMS to keep track of analyses, periodically checking sample status to determine whether data is being entered in a timely fashion. In addition, SIMS and associated programs allow the senior staff to rapidly examine data for consistency among samples and institute appropriate QA measures.

When analysts have completed a test or series of tests on a group of samples, data are entered into the computer. Once all of the data have been entered, they are examined by a senior staff member for consistency and verified. When all tests scheduled for a particular sample are completed and entered, SIMS removes the sample name from the active sample index, and adds the name to the sample archive index.

Additional aspects of the SIMS system which promote smooth sample flow are: (a) the production of a sample backlog list once a week that lists all samples which approach a storage expiration date; and (b) a test backlog list for specific analyses also on a weekly basis, to keep the analysts aware of the samples which have not been completed. Thus SIMS acts as an overall coordinating system for analysis and quality assurance. It even offers the opportunity to provide comments on a particular sample (for instance a preservative problem or a QA comment).



sympa lengthera lengther biblishs services besides besides reacted reacted respect levels (1925)

FIGURE 2

V. CHAIN OF CUSTODY

A. Introduction

A written record of sample possession is required for all samples whose analytical results may be used in a court of law. The essence of chain of custody procedures is to provide evidence that a sample has not been adulterated. The primary objective is to create an accurate written record tracing the possession of the sample from collection through its introduction as possible court evidence.

Custody consists of a sample being in actual physical possession or locked up to prevent unauthorized access.

B. Rules for Sample Collection

- 1. Obtain samples using appropriate sampling techniques.
- 2. Transfer custody of the samples as little as possible.
- 3. Attach a chain of custody record bottle tag to the sample container at the time of sample collection. The tag contains information on sample number, date, time, source, analyses required, name of person doing sampling, and witnesses. The tag is signed and dated (including time) by the sample collector. The sample container is sealed so that the bottle cannot be opened without breaking the seal.
- 4. Field measurements are recorded with ink in a bound field notebook or log. A separate set of field notebooks are maintained and stored in a safe place where they can be accounted for at all times. Entries are signed by the person taking the sample and errors crossed out with one line and initialed.
- 5. The person taking the sample is responsible for the care and custody of the sample and must assure that each container is in his physical possession or view at all times or is stored such that it cannot be tampered with.

C. Transfer of Custody

When transferring custody the transferree must sign and record the date and time on the chain of custody record tag. The field custodian is responsible for properly packaging and dispatching samples to the appropriate laboratory for analysis.

All samples are accompanied by a sample transmittal form which includes information identifying the contents. If samples are delivered to the lab when appropriate personnel cannot receive them they must be locked in a designated area so that no one can tamper with them.

D. Laboratory Custody Procedures

The principal of "Chain of Custody" is that samples are handled by a minimum number of persons.

Two full time employees are designated as sample custodian and alternate for sample handling. In addition the lab has a clean dry isolated area that can be securely locked from outside as a "Sample Storage Security Area". The sample custodian must maintain a bound log book to record appropriate information about such samples.

Immediately upon receipt the custodian affixes a number to the attached tag, records the required information in the log book and preserves the sample if that has not already been done. The sample is then stored in a locked sample area.

Lab personnel are responsible for the care and custody of the sample once it is handed over to them. Once the sample testing is completed the unused portion of the sample along with any identifying tags and seals are returned to the custodian.

VI. GENERAL PROCEDURES

Prior to the collection of a sample, the collector is briefed by a Section Head or QA officer as to proper sampling, transportation, and preservation procedures.

A. Sampling

- 1. Recommended sample containers, minimum sample volumes, preservatives, and maximum holding times are listed on the laboratory sheet entitled "Collection, Preservation, and Storage of Water Samples" (Attachment 1). Sampling procedures follow the guidelines established by the appropriate discharge permit, Standard Methods, EPA, and the senior staff recommendations.
- 2. Sample identification. The following information is required on each sample submitted. This information is to be written on the container with a permanent felt tip marker.
 - a. Client and/or job number
 - b. Sampling date
 - c. Sample I.D.
 - d. Analyses required

In cases where chain of custody is required, the appropriate forms and labels are obtained from the QA officer.

B. Accepting Samples

The Log-in Clerk inspects a sample upon arrival. If the integrity of the sample has been affected, the person requesting the analysis is

contacted. If the sample is satisfactory, then it is logged into SIMS with the following information:

- 1. Laboratory Number
- 2. Client
- 3. Address
- 4. Job Number or P.O. number
- 5. Person requesting analysis
- 6. Test(s) requested
- 7. Date received
- 8. Date collected
- 9. Person collecting the sample (if known)

C. Labeling of Reagents

Whenever a new bottle of a reagent arrives, the date received is to be stamped on the label.

Whenever a new bottle of a reagent is prepared, a container label with the following information is attached:

- 1. Reagent and concentration
- 2. Analysis for which the reagent is intended
- 3. Date prepared
- 4. Initials of person preparing solution

Data for reagents which require standardization are recorded in the Standardization Log. It is vitally important to record this information in the log along with the initials of the analyst performing the standardization. The section head and QA officer review this log quarterly.

VII. QUALITY CONTROL - INORGANIC CHEMISTRY

A. Concepts

THE PARTY OF THE P

The QA/QC program for laboratory work is based on the EPA guidelines for quality assurance. The core of the program is a properly trained and experienced staff integrated with a system developed to discover analytical discrepancies. There are four central aspects to our QA/QC program.

- 1. Field blanks are prepared wherever possible or if mandated by regulations or client.
- 2. Checks on instrument performance are done on a routine basis.
- 3. Replicates, spikes and external standard reference materials are used in ten percent of the sample load to assure acceptable data.
- 4. A verification test on all sample sets ensures that final data are screened for consistency and accuracy by a responsible analyst.

B. Routine QA/QC Program

All instruments have a routine maintenance program which is followed on a regular basis to insure optimum performance. This maintenance involves checking electronic response, detection limits, and behavior on standard solutions. It may also entail cleaning glassware in the instrument on a regularly scheduled basis. For example, the ICP glassware (nebulizer and torch) is cleaned after every 8-10 hours of use and performance for manganese is checked to determine that the appropriate sensitivity and precision are being acheived. Only when these checks are completed is a series of samples analyzed.

All of the instrumental analyses are subject to the checks summarized below:

- 1. Verify proper functioning of instrument mechanics.
- 2. ICP check performance on Mn
- 3. CFA check reagents, standards
- 4. UV/VIS check performance on standards
- 5. IC check resolution of standard solution, check peak height on standard solution.
- 6. AAS check performance of standard solutions

Results of these checks (and deviations) are entered into the instrument logbooks.

During an analytical run duplicates are analyzed with a frequency of no less than every tenth sample. The comparison between duplicates is used as a further guide to QC data. Based on an initial series of duplicates quality control limits are established using Shewart control charts (Attachment 2). When duplicate results fall outside the control limits all data between duplicates are examined to locate the problem. Once the problem is located, the samples are reanalyzed. In addition, the control charts allow determination of long-term trends which may suggest a more subtle analytical bias.

In addition to duplicates, blanks and standards are analyzed at least every tenth sample to check for drift or contamination which may degrade the quality of the results.

Several additional QC measures are applied to insure optimum results. Whenever a sample is analyzed for enough parameters to perform a cation-anion balance (Ca, Mg, Na, K, Cl, SO₄, NO₃, F, Alkalinity), the balance is checked to insure that it falls within a range of 2-5 percent, depending on the sample source. (For treated waters we strive for ±2 percent, for raw and waste waters ±5 percent although 2 percent is usually achieved).

The table below summarizes various tests for which alternative procedures are available and used routinely as analytical performance checks.

Parameter
Anions (Cl, F, SO₄, NO₃)
Alkalinity
Ca, Mg
Other Metals

Cross Checking Method
CFA and IC
CFA and Manual Titration
AAS and Manual Titration
AAS and ICP

All of these approaches make up our QA/QC program. Only when data have passed all these checks does a senior staff member verify data for a sample and permit it to be transmitted to the VAX and the client.

C. Glassware

STORING PROPERTY

TO TOTAL STATES STATES TO THE TANK STATES ST

The metals, glassware, and bottles are washed in the following manner:

- 1. Normal detergent wash in a dishwasher which includes rinses with tap and deionized waters.
- 2. Rinse with DI water
- 3. Soak in 4N HNO3 overnight.
- 4. Rinse five times with deionized water.

This acid washed glassware is stored in the drawers marked "Acid Washed Glassware". Following step (3) metals glassware is handled only with polyethylene gloves.

The normal glassware washing procedure includes only (1). If there are organic residues left on the glass, the sulfuric acid - No chromix acid treatment is to be used. For low level phosphate determinations, glassware should be soaked in 6N HCl and rinsed with freshly deionized water.

Calibrated glassware, such as volumetric flasks and pipettes should be completely dry and cooled to room temperature before being placed in their respective places. They are not to be oven dried.

Odor analysis flasks are acid-washed, rinsed and baked at 400°C prior to use.

D. Instruments

On an as-needed basis, instruments must be serviced and checked out according to the instruction manuals. If an instrument is not performing according to specification, the appropriate instrumentation specialist is to be called.

All instruments have an instrumentation log with maintenance information filled out on a routine basis. All analysts working with a given instrument must familiarize themselves with the routine maintenance and performance checks for that instrument. Failure to adhere to performance checks can seriously affect the accuracy of results.

E. Reagents

The routine standardization of reagents for titrations serves as the primary method for maintaining accurate analytical results in titrimetric analyses. These results and the initials of the analyst doing the tests are recorded in the <u>Standardization Log</u>. If there is a noticeable discrepancy, the senior staff should be consulted. If problems exist as evidenced by the data, they must be resolved before any further analyses are performed.

A standard should be analyzed along with every set of samples to verify accuracy of end-point determinations.

Reagents and stock standards for colorimetric or instrumental analyses reactions are labeled with the date and analysts' initials. Data and calculations are recorded in the laboratory notebook for that analysis.

Stock standards and reagents have holding times as noted below.

- 1. Metal Stocks:
 - a) >10 mg/L 6 months
 - b) 1-10 mg/L 60 days
 - c) < 1 mg/L fresh
- 2. Anions, Nutrients, Cyanide, Phenol, and COD Stocks:
 - a) 60 days until longer holding times are demonstrated.
 - b) Working standards are made up fresh if specified by the method.

When a new stock standard is prepared, it must be cross-checked against the old one. Stock standards must contain specified preservatives and conform to specified methods of storage.

F. Verification of Standard Curve for Spectrophotometer Analyses

- 1. Before use, subsequent standard curves are verified by use of at least one reagent blank and one standard at midrange of the working curve. The values obtained must be within +10 percent of the original curve.
- 2. If twenty or more samples are to be analyzed, the working standard curve is verified by running an additional standard at midrange every ten samples. These checks must also be within +10 percent of the original curve.
- 3. Absorbance values for the standard curve are recorded in instrument logbooks.

G. Verification of Precision and Accuracy

One duplicate sample and spike is run every ten samples, or with each set of samples if there are less than ten samples analyzed. Checks should be within +2 standard deviations and are plotted on the control charts (see below).

H. Quality Control Charts

Our QC charts are based on procedures outlined in the attached excerpt from the EPA Manual (600/4-79-020). The QC charts are kept up to date by the analyst performing each test. All data must be available for inspection by senior staff and the QA officer.

When insufficient analyses are performed (eg., monthly) to establish parameters for the QC charts, data is confirmed by duplicates and spikes is possible. A guide to expected precision for a number of analyses is found in the ASTM Manual on Water Analysis.

I. Quality Control - Heavy Metals

KASSASA IZZZZZZ IKASKAS SZZZZZK SZZZZZR SZZZZZR KASKAS KASKAS KASKAS

- 1. Initially a calibration curve composed of at least a reagent blank and 2 standards is prepared. When it is certain that results are within the linear range, one standard is used for flame A.A. Subsequent calibration curves are verified by use of at least a reagent blank and one standard at or near the maximum contaminant level (MCL).
- 2. If twenty or more samples are analyzed per run, the working standard curve is verified by running an additional standard within the range of sample values after every tenth sample.
- 3. Reagent blanks are run for each metal analyzed at a frequency of 10 percent with the sample values being corrected accordingly.
- 4. At least one duplicate sample is run after every sample, or with each set to verify precision of the method. Checks should be within the control limit established by the QC charts.
- 5. Spiked aliquots are analyzed with a frequency of -10 percent of the sample load. If the recovery is not within ±10 percent of the expected value, the sample is analyzed by the method of standard additions. Whenever a new complex matrix is analyzed either samples and standards are matrix matched or the sample analyzed by standard additions.
- 6. A known reference sample is analyzed with every run to verify the accuracy of results.

- 7. When using the method of standard additions, a linear curve over the entire range of addition is necessary for the results to be considered valid.
- 8. When using the furnace, negative peaks caused by interferences are not used in calculations. The sample should be diluted 1/10 and if necessary 1/10 again until the negative deflection is eliminated.
- 9. Unknown performance samples are analyzed once per quarter.

 Results must be within the control limit. If problems arise, they are corrected, and a follow-up performance sample is analyzed.
- 10. A standard at the MCL is analyzed along with the samples whenever drinking water is analyzed.
- 11. Bottle blanks are analyzed monthly to monitor glassware cleanliness. These blanks consist of DI water stored in a randomly chosen metals bottles.

J. Quality Control - Odor Analysis

Company to the contraction of th

MANAGER CANADANA SANGERS

- 1. All glassware for odor analysis is acid washed, baked at 400°C, and rinsed with odor-free water two times before daily use.
- 2. Glassware used for a sample is not to be used more than once without acid-washing and baking if a TON of 40 or greater is measured.
- 3. Odor-free water is prepared fresh each day an odor analysis is run.
- 4. Between samples, glassware is rinsed with odor-free water prepared by activated carbon filtration of deionized water.
- 5. An odor-free water blank is carried through with each set of samples analyzed.
- 6. At the beginning of a series of odor analyses, four randomly chosen flasks are carried through the procedure as blanks. If any of these have a noticeable odor, the entire lot is checked and the rejected flasks rewashed.
- 7. At least one sample per day is analyzed in duplicate on two different runs.

K. Anion-Cation Balance

The percent error for ion balance is described in the following equation:

$$\% error = \frac{\sum cations - \sum anions}{\sum cations + \sum anions} \times 100\%$$

where:

anions = anions in milliequivalents (A)
cations = cations in milliequivalents (C)

When the percent error exceeds 2 percent on finished waters (5 percent on raw waters), the analysis is rechecked.

For low level samples the acceptable percent error is defined as:

$$\frac{10.65 \times 2 + 1.55(\Sigma C + \Sigma A)}{2(\Sigma C + \Sigma A)} \tag{1}$$

Equation (1) is derived from Standard Methods.

If the percent error is exceeded, the constituents to be rechecked are determined in the following fashion:

THE STATE OF THE S

- 1. Divide the Specific Conductance by 100.
- 2. Determine the difference between this value and the Σ anions and Σ cations. The larger difference determines the group of ions to be checked.
- 3. The ion having the largest meq/l should be checked first. If there is still too large an error, the ion having the next largest milliequivalency is checked, etc. until a satisfactory balance is achieved.

L. Comparative Cross-Checking of Methods

Whenever alternative analytical techniques are available, results from random samples are cross-checked.

If results on reference samples differ by more than 10 percent, the analyses are checked (e.g., standards and reagents should be rechecked and analyses should be cross-checked by alternate methods).

These data are used to provide comparability data for non-standard methods in addition to checking accuracy.



and the second of the second of the second

VIII. QUALITY CONTROL - ASBESTOS

A. Personnel

All are experienced or trained by an analyst with 2 years minimum experience in asbestos determination.

B. Records

SE LESSONAL MARKERS DESSESSE LESSESSES LESSESSES ABOUT MARKERS

All quality control data is available for inspection upon request. Worksheets are maintained for rechecking calculations. All permanent records are kept in bound volumes.

C. Sampling Procedures

The sampling technique follows the methods outlined by the EPA on page 3 of the <u>Interim Method</u> for <u>Determining Asbestos</u> in <u>Water EPA-600/4-80-005</u>, January 1980. All samples are to be stored at 4° C until filtration and completion of analysis.

D. Measurements/Analyses

The standard accepted procedure is outlined in the <u>Interim Method</u> previously mentioned. All modifications of procedures including reasons for modifications are recorded.

E. Reports

All calculations and reports are entered into the lab computer to eliminate inconsistency in the final report. The analyst and the immediate supervisor must check and then sign the final report before sending it to the client.

F. Instruments

The manufacturers' manuals for proper operation of all equipment used in asbestos analyses are properly filed and accessible. Records of periodic inspection, calibration and service of equipment is maintained in appropriate log books. Phone numbers for instrument service are posted by each piece of equipment.

G. Quality Control

- 1. All materials used in the analysis are dated upon receipt and replaced as needed or when shelf life has expired.
- 2. A negative control blank using fiber-free water is processed each day that samples are filtered as stated on page 23 of the <u>Interim Method</u>. Two hundred ml of asbestos-free water must have no more than 2 fibers in 20 grid squares of a 200 mesh grid.

- 3. Standard asbestos fibers are checked twice a month (or as required) for identification, EDX performance, standard morphology photographs and diffraction patterns.
- 4. A duplicate sample is run at least once per ten samples processed.
- 5. Once a year a sample is split with the EPA or another approved asbestos laboratory.
- 6. The lab participates in approved round-robin asbestos test evaluations (when available).
- 7. The lab analyzes a reference sample or an unknown performance sample once per year.
- 8. Analytical results are cross-checked before the final report is sent to the client.
- 9. All samples are filtered within 48 hours of their arrival.
- 10. The absolute (HEPA) filtration system is monitored daily.
- 11. Asbestos glassware is prepared as stated in the EPA Interim Method on page 12.
- 12. When several grids of the same sample are counted, the deviation should not exceed +15 percent as stated on page 27 of the EPA Interim Method.

IX. QUALITY CONTROL - RADIOCHEMISTRY

- 1. All QC data are available for inspection to determine validity of laboratory results.
- The laboratory participates twice each year in an appropriate EPA-administered performance study on unknowns. Results must be within the control limits established by EPA for each analysis. If results are outside these limits, efficiencies are immediately rechecked.
- 3. Counting-instrument operating manuals and calibration protocols are available to analysts and technicians.
- 4. Calibration data and maintenance records on all radiation instruments and analytical balances are maintained in a permanent record.

5. Minimum Daily QC

- a. To verify precision of methods, a minimum of 10 percent of the samples are duplicates. Checks must be within +2 standard deviations of the mean range.
- b. A background sample is run each day radiation samples are analyzed.
- c. Quality control performance charts or performance records are maintained.
- d. Reported results include the 2 sigma error for counting.
- 6. Plateau curves and efficiency checks are run quarterly and the data recorded in the radiation notebook.

X. QUALITY CONTROL - MICROBIOLOGY - PASADENA

A. Sampling

experses gravered bases over the contraction of the contraction and contraction and contraction to the contraction of the contr

The individual collecting samples should be aware of the sampling precautions outlined in <u>Standard Methods</u> and summarized below. These precautions maintain the integrity of the samples.

- 1. Prevent contamination of the sample bottle by collector.
- 2. Prevent contamination from other sources not representataive of sample, e.g., rust from sampling tap and sediment from stream bed.
- 3. Do not rinse the sample bottle with the sample.
- 4. Leave 1/2" headspace in the container and cap tightly.
- 5. Identify and describe the samples as completely and accurately as possible. This information will aid the analyst in determining the dilutions to inoculate. The following information is to be provided:
 - a. Client
 - b. Sample date
 - c. I.D. number
 - d. Analyses requested
 - e. Water type and/or source

B. Sample Storage and Shipment

If the samples cannot be analyzed within 4 hours, they are stored on ice in a cooler. First, the sample bottles are checked to insure tight caps and no leakage. The bottles are then wrapped in a strong plastic

bag and sealed. This keeps the melting ice from contaminating the samples. Ice cubes or their equivalent are placed around the samples. Care must be taken that the samples do not freeze. Package the cooler securely for shipment.

The samples must reach Pasadena within 30 hours in order that they fall within the maximum holding time.

C. Steam and Dry Heat Sterilization

1. Steam Sterilization:

- a. On the front of the autoclave are a set of instructions for the operation of the Market Forge autoclave. The lab assistant should note that the timed cycle does not start until the pressure reading is 15 psi and temperature is 121°C. The material, time, pressure, and temperature are recorded in the temperature Log book.
- b. Sterilization indicators, heat sensitive tape, steam clocks, or discs, are available and are used during each cycle. If problems exist as indicated by a failure of one of the various sterilization indicators, consult with a senior staff member.

D. Precautionary Procedures and Practices

- 1. Media preparation: Only demineralized water is used for the preparation of media.
- 2. Media storage: Once opened, the powdered media is stored top down to prevent hydration of the medium.

Prepared liquid medium is stored at 4°C. The basket should be labeled with the type of medium, date and the person who made it up.

Prepared agar plates are stored in plastic bags, agar up, in the refrigerator. The bag is labeled to identify the type of medium, date made, and person who prepared it.

3. Incubator operation: On a daily basis when bacteriological samples are incubated in a water bath or incubator, the temperature is recorded each morning in the appropriate log. The temperature can also be checked with the NBS calibrated thermometer at 20°C and 35°C. A thermometer calibrated at 44.5°C is used when fecal coliforms are incubated.

E. Quality Controls

- 1. Positive and negative controls for MPN are used each time the analysis is performed. Positive controls utilize a known coliform such as <u>E. coli</u> or <u>E. aerogenes</u>. Negative controls are sterile phosphate buffer.
- 2. The completed test is conducted on 10 percent of the positive drinking water samples. If no positives are found, at least one positive source water is completed quarterly. Both gram(+) and gram(-) controls are used for the gram stain.
- 3. The lab analyzes a bacteriological reference sample from the California State Department of Health annually. An MPN test, through the confirmatory step and standard plate count, is conducted on this reference sample.
- 4. Media supplies are dated upon receipt and when initially opened. A written record is maintained on media. When receiving new media the date, the type of media, source of media, and initials of the person logging in the media is recorded in the Media Log book.
- 5. Media prepared in the lab is logged into the Prepared Media Log by the laboratory assistant. These records include media lot number, date, sterilization time and temperature, final pH, and the assistant's initial.
- 6. Operating temperatures of incubators, waterbaths, hot air ovens, and refrigerators are checked daily, logged in the maintenance book and adjusted if necessary.
- 7. Autoclave temperatures and pressures are logged in the maintenance book for each cycle of use. Sterilization indicators are used each time the autoclave is operated.
- 8. Quality of laboratory pure water is analyzed monthly and annually as described below:
 - a. Monthly—conductivity, pH, TOC, chlorine residual, and plate count
 - b. Annually-bacteriological suitability test, trace metals (Pb, Cd, Cr, Cu, Ni, and Zn).

Criteria

pH 5.5 to 7.5
EC <5 \mmhos/cm
Trace Metals <5 ppb each
Bacteriological 0.8 to 2.0
Suitability Ratio

- c. Data is recorded into the Quality Assurance Log.
- 9. The laboratory distilled water is tested annually for bacteriological quality as described in Standard Methods.

The washing and sterilization procedures for laboratory glassware are checked annually by testing glassware for inhibitory residues.

F. Ames Salmonella/Mammalian-Microsome Mutagenicity Test

- 1. Quality Control Check for Mutation Integrity of Tester Strains
 - a. The presence of the RFA character and R factor, along with histidine requirement and spontaneous reversion are tested during each mutagenicity test series and when a new frozen permanent is used according to the methods of Ames.

Acceptable spontaneous reversion counts are in the following ranges: 10-34 (TA 1434), 3-15 (TA 1537), 30-50 (TA 98) and 120-200 (TA 100).

- b. Reagents and media are stored at 4° C for no more than two weeks.
- 2. Positive and Negative Controls

NOTESTAN TACESTAN STREET, STREET,

a. Three compounds are used as positive mutagen controls for the four bacterial strains. A dose-response curve is made for each strain exposed to a mutagen during each test series.

Positive Controls

Standard Mutagen	<u>Strain</u>
2-Aminofluorene	TA98 (+S-9)
Sodium Azide	TA1535
Methyl methansulfanate	TA1537, TA100

b. Negative controls are run on reagents and media used in the test: These include sterility tests for dimethyl sulfoxide, S-9, the test compound and agar media. In addition,

the absence of mutagenic activity is confirmed for dimethyl sulfoxide.

G. Endotoxin Assay

- 1. Sample Collection and Preparation
 - a. Samples are handled aseptically with pyrogen-free glassware. All glassware is depyrogenated in a dry hot air oven at 400° C for 1.5 hours.
 - b. Fresh distilled dilution water is depyrogenated in an autoclave for 1 hour at 121.50 C.
 - c. Pyrogen-free NaOH or HCl are used to adjust samples to a pH range of 6-8.

2. Reagent Storage and Stability

- a. Limulus lysate, positive control and negative control are stored according to the manufacturer's recommendations. Expiration dates are marked on the vials containing the reagents. Unused vials remaining after expiration are discarded.
- b. The lot number of each group of lysate and controls is recorded for each test series.

3. Initial Quality Control Procedure

The following is a description of the procedure used by the analyst:

- a. Before any tests are performed, the variability of the test is assessed. A minimum of 4 and a maximum of 28 tests are performed by the analyst using a single lot of lysate and a single lot of endotoxin. For each group of 4 replicate tests a single series of dilutions of the endotoxin is prepared. A twofold dilution series is used with a range suitable for yielding an end-point with the lysate being tested. After incubation, the reaction in each tube is recorded as either positive or negative. The end-points are expressed as ng/ml and then converted to log2 and the standard deviation (S.D.) calculated.
- b. If the S.D. is less than or equal to the value for the 99 percent limit on the S.D. for 4 samples (see following table), the test is in control. If the S.D. is greater than the tabulated value, the test may be expanded in increments of 4 tests up to the maximum of 28 tests and a new S.D. calculated using data from all tests. If after doing 28 tests the S.D. still exceeds the 99 percent upper limit value

corresponding to 28 tests, the tests are invalid due to excessive variability. If after any set of 4 replicates, the S.D. is equal to or less than the tabulated value for that number of tests, it can be concluded that the variability is under control and no additional tests are necessary. At that point the geometric mean (GM) and its 95 percent limits of end-points from all tests performed are calculated. If the indicated value for the lysate used falls within these limits, the tests have confirmed the label claim. If it is not, the source of error is determined before doing additional testing.

99% Upper Limit on S.D. of Log₂ (Lysate Sensitivity)

Sample Size	99% Upper Limit for S.D. (Log ₂ L.S.)
4	*1.77
8	1.48
12	1.36
16	1.30
20	1.26
24	1.23
28	1.20

^{*}Limits can be converted to log_{10} by multiplying each value by 0.30103.

H. Mammalian Cell Transformation Assay

- 1. Cell Culture Quality Control
 - a. Only published cell culture methods are used.
 - b. Whenever conditions change, or a new batch of medium or serum is used, the growth rate and plating efficiency of the cultures are monitored.
 - c. Cells are grown in a humidified 5 percent CO₂ incubator where temperature and CO₂ can be closely monitored.
 - d. No antibiotics are used in any of the cultures since they can mask chronic infections or cause the emergence of bacterial L-forms.

I. Parasites

SCECCES SEPTIMENTS SEPTIME

STATE STATE OF THE STATE OF THE

THE PROPERTY SECTIONS

Quality control in parasitology is not as well defined as other areas of microbiology. Sample collection and processing quality controls are similar to other microbiology assays with respect to maintaining the integrity and stability of biological materials. This is accomplished by

refrigerating the orion filters after the completion of sampling until they are processed for parasite identification.

Filters are stored in polypropylene zip-lock bags. Upon receipt, a 10 percent formalin fixative solution is added to the bag containing the filter.

J. Enteric Viruses

STREET, STATE OF THE STATE OF T

ACCURATION INCIDENCE INCID

1. Sample Integrity

During all phases of enteric virus monitoring, the samples must be protected from external and cross-contamination. Contamination from the equipment, the laboratory environment, and personnel handling the samples is prevented by adhering to the following practices:

- a. Equipment used to filter and elute enteric viruses at the sampling site are disinfected with a strong hypochlorous acid solution (100 ppm Cl) after each sample run. The chlorine contact time is 10 minutes. Equipment is rinsed with a sodium thiosulfate solution after being rinsed thoroughly with tap water.
- b. When several sites a day are sampled with the same equipment, the cleanest site, such as chlorinated effluent from a water treatment plant, is sampled first with progressively dirtier samples following in order.
- c. Manipulation of the sample is temporally spaced so that only one step proceeds in the laboratory at a given time. The order is generally from the dirtiest procedures to the cleanest ones.
- d. Manipulation of the samples is spacially separated in the laboratory such that reconcentration of the filter eluent takes place in a different room from inoculation of the cell monolayers. Preparation of the media and cell monolayers is conducted under a laminar flow containment hood to prevent bacterial and fungal contamination. Antibiotics are incorporated into the monolayers to minimize this type of contamination.
- e. Contamination of laboratory surfaces is prevented by the use of ultraviolet disinfecting lamps in the ceiling which are turned on in the evening after all personnel have left the area.
- f. Each cell line is worked with separately to prevent cross-contamination of cells.

XI. QUALITY CONTROL - ORGANIC CHEMISTRY

A. Sampling

- Sampling instructions for each of the organic analyses accompany all sampling containers. The instructions meet or exceed the requirements of <u>Standard Methods</u> and the EPA and completely describe sampling, preservation and shipment precautions.
- 2. Field travel blanks are taken according to regulations or as requested by the client. It is recommended that one travel blank for each type of analysis be included with each set of samples taken.

B. Standard Preparation

All standards are prepared gravimetrically on at least a quarterly basis, or more often as required. They are prepared gravimetrically. If the chemical being weighed out is a possible or known animal carcinogen then it is weighed out in the carcinogen hood. Calculations and details are recorded in the Organics Standards Lab notebook and in the appropriate Quality Control notebook. Stock standard solutions are stored in organic free 3 ml amber vials with teflon seals. Each vial is labeled with the date, standard I.D., and the preparer's name. Stock solutions are stored at -20°C until use. Old standards are disposed of as hazardous waste after the new standard has been cross checked and properly verified.

C. Quality Control - GC/MS

CONTROL SECRECE DESIRENCE CONTROL CONTROL CONTROL SECRETARY PROPERTY SECRETARY SECRETARY SECRETARY

1. GC/MS Tuning and Specifications

a. The mass spectrometer must maintain proper mass fragmentation characteristics. This is done by running either BFB or DFTPP and ensuring that the spectrum meets the specified criteria. Copies of the spectral list are entered in the GC/MS Quality Control book once per week at a minimum. Samples are not run unless BFB or DFTPP spectra can be produced which meet the specified criteria.

DFTPP Key Ions and Ion Abundance Criteria*

Mass	Ion Abundance Criteria
51	30 to 60 percent of mass 198
68	Less than 2 percent of mass 69
70	Less than 2 percent of mass 69
127	40 to 60 percent of mass 198
197	Less than 1 percent of mass 198
198	Base peak, 100 percent relative abundance
199	5 to 9 percent of mass 198

275	10 to 30 percent of mass 198
365	Greater than 1 percent of mass 198
441	Present but less than mass 443
442	Greater than 40 percent of mass 198
443	17 to 23 percent of mass 442

BFB Key Ions and Ion Abundance Criteria*

Mass	Ion Abundance Criteria
50	20 to 40 percent of mass 95
75	50 to 70 percent of mass 95
95	Base peak, 100 percent relative abundance
96	5 to 9 percent of mass 95
173	Less than 1 percent of mass 95
174	70 to 90 percent mass 95
175	5 to 9 percent of mass 95
176	70 to 90 percent mass 95
177	5 to 9 percent mass 95

- (1) The following settings are maintained:
 - o Mode Direct

THE PARTY OF THE PROPERTY OF THE SECOND PROPERTY OF THE PARTY OF THE P

- o Emission Current: 0.5 ma
- o Electron Energy: 70 eV
- o Electron Multiplier: 1000-2000V as necessary
- o Dynodes: 3000V
- o Ion Source: 300°C
- (2) The GC/MS is calibrated daily with FC43 gas to obtain a millimass defect ≤20 millimass units. The calibration is verified with the FIT program to an RMS error of ≤2 percent.
- (3) The instrument zero is checked using the zero control and adjusting as necessary to provide a minimum background of electrical noise.
- 2. GC/MS Analytical Quality Control (Base-Neutral and Acid Extractables, Closed-Loop Stripping, MIB/Geosmin, Volatiles).
 - a. For all analyses a laboratory water blank is analyzed to check for artifacts from the GC/MS system and for the presence of impurities in the water blank making it unsuitable for standard preparation and sample dilution.
 - b. Field blanks are analyzed on a routine basis as a part of the quality assurance program. With each set of travel blanks sent out, a stationary travel blank is kept in the laboratory for analysis to demonstrate that the water sent out was free of contamination.



- c. Once a week a three point standard curve is run for each type of analysis being performed. These curves are used for comparison with the daily calibrations. A plot of each standard curve is kept in the GC/MS Quality Control notebook.
- d. Standards are analyzed for each GC/MS analysis daily. Proper instrument sensitivity and stability are determined by comparison of performance with earlier weekly calibration curves. Significant deviation from previous quantitation curves requires running a complete new set of calibration standards.
- e. The precision and accuracy of the technique is determined by running 10 percent of the samples either in duplicate, or with a standard spike addition. The results of the duplicates and the spikes are kept in the GC/MS Quality Control notebook.
- f. Blind check samples are purchased from outside laboratories and analyzed on at least a quarterly basis. EPA check samples are analyzed when available and the results compared to the published results. All results from these check samples are kept in the GC/MS Quality Control notebook.
- g. When each new GC/MS stock standard is prepared, the previous standard and the new one are analyzed in triplicate. If the difference between them is greater than 10 percent, another standard is prepared. The results of these analyses are kept in the GC/MS Quality Control notebook.

- h. Instrument maintenance is noted in the GC/MS Quality Control notebook. This includes source cleaning, column changes, and similar routine maintenance.
- i. A plot of the average internal area count for each analysis for each day is kept in the GC/MS Quality Control notebook.

D. Quality Control - Gas Chromatograph

25.77.8

SERVICES LINESPEND LINESPER LONDON CONTINUE

- 1. For all analyses a laboratory water blank is analyzed to check for artifacts from the GC system and for the presence of impurities in the water blank making it unsuitable for standard preparation or sample dilutions.
- 2. Field blanks are recommended and are provided and analyzed when requested. With each set of travel blanks sent out, a stationary travel blank is kept in the laboratory for analysis to insure that the water was free of contamination.

- 3. Once a week a standard curve is run. For trihalomethane analysis this is a seven point curve, for Pesticides/PCB/Herbicides analysis this is a three point curve. These curves are used for comparison with the daily calibrations. A record of each standard curve is kept in the GC Quality Control notebook.
- 4. Standards are analyzed daily for each routine analysis. Standards are extracted along with the samples to ensure adequate recovery. Proper instrument sensitivity and stability are determined by comparison of performance with earlier weekly calibration curves. Significant deviation from previous quantitation curves not directly attributable to instrument adjustment requires running a complete new set.
- 5. The precision and accuracy of the technique is determined by extracting 10 percent of the samples either in duplicate, or with a standard spike addition when enough sample is provided. The results of the duplicates and the spikes are kept in the GC Quality Control notebook.
- 6. Blind check samples are purchased from outside laboratories and analyzed at least on a quarterly basis. EPA WS series check samples are run and the results compared to the published results. All results from these check samples are kept in the GC Quality Control notebook.
- 7. When each new THM or Pesticide/PCB/Herbicide stock standard is prepared, the old standard and the new one are analyzed in triplicate. If the difference between the means of these triplicates is greater than 10 percent another standard is prepared. The results of these analyses are kept in the QC Quality Control notebook.
- 8. Any maintenance to the instrument is noted in the GC Quality Control notebook.
- 9. A record of the average internal area count for each analysis set utilizing an internal standard is kept in the GC Quality Control notebook.

E. Quality Control - Total Organic Halogen (TOX)

para increases haracter latinations l'executes espaines ambandes

- 1. The analyzer module is run with the boat empty at the beginning of each day to remove all contamination accumulated since previous analyses.
- 2. The response of the titration cell is checked each day by injecting 10 µg of inorganic chloride directly into the titration cell solution. Injections are repeated and adjustment of the instrument made until two subsequent replicates within 2 percent are obtained.

- 3. Duplicate 10 ug organic chloride instrument calibration standards (standards injected directly onto the carbon contained in the adsorption columns) are analyzed at the beginning of each day. One additional replicate of 10 ug organic chloride is analyzed for every 10 samples analyzed. These results are kept in the TOX Quality Control notebook.
- 4. Triplicate carbon blanks (carbon packed adsorption columns washed with nitrate-wash solution only) are analyzed at the beginning of each work shift, and subsequently one blank is analyzed after each 10 sample analyses. These results are kept in the TOX Quality Control notebook.
- 5. All samples are analyzed in duplicate. If the net values of the duplicates are not within 10 percent of one another, a third replicate is analyzed. The absolute difference between one in every ten duplicates is plotted on the Shewhart plot which is appropriate for that analysis and sample value. These plots are kept in the TOX Quality Control notebook.
- 6. The titration cell is revitalized by rinsing with fresh cell electrolyte after approximately every twenty analyses. After revitalization of the titration cell, its stability and response are checked by an injection of the inorganic chloride standard.
- 7. The volume of sample filtered is adjusted for the sample analyzed to produce adsorbed organic halogen content within the range of optimum function of the instrument (5-40 µg).
- 8. Once during each work shift, the top and bottom adsorption columns from one sample adsorption are analyzed separately to determine if any organic halogen breakthrough is occurring. These results are kept in the TOX Quality Control notebook. Top and bottom adsorption columns are run separately on all samples with high levels of TOX or high concentrations of inorganic halide.
- 9. Every twentieth sample is spiked and recoveries are determined.
 These results are kept in the TOX Quality Control notebook.
- 10. The purity and adsorption capacity of each new batch of carbon purchased is assessed by analysis of seven replicates each of reagent water, carbon blanks, instrument calibration standards, and adsorption efficiency standards (standards injected into reagent water then filtered). The results of these analyses are to be kept in the TOX Quality Control notebook.
- 11. Quarterly, a seven point standard curve is run by spiking reagent water with seven different concentrations of organic halogen. This curve is kept in the TOX Quality Control notebook.

NACKACK INTERNAL (SERVICE LONGER MANAGE

- 12. Each week a three point standard curve is run by spiking reagent water with three different concentrations of organic halogen. This curve is kept in the TOX Quality Control notebook.
- 13. Each day one standard is run in duplicate. The average of this standard must be within 10 percent of the value for that standard on the weekly standard curve or a new standard curve must be run.
- 14. Any maintenance to the instrument is noted in the TOX Quality Control notebook.
- 15. When each new TOX stock standard is prepared, the old standard and the new one are analyzed in triplicate. If the difference between them is greater than 10 percent another standard is prepared. The results of these analyses are kept in the TOX Quality Control notebook.

F. Quality Control - Total Organic Carbon (TOC)

TO STATE OF THE ST

LOCACERS INCHERENCE INCHESSES INCHESSES SECTION

- 1. Prior to analysis of samples an instrument calibration standard is analyzed in order to update the instrument calibration factor and to determine instrument performance. The uncalibrated instrument response factors are recorded in the TOC Quality Control notebook.
- 2. Between each ten samples at least three standards and the laboratory water blank are analyzed in duplicate.
- 3. All samples are analyzed in duplicate. If the net values of the duplicates are not within 10 percent of one another, a third replicate is analyzed. The absolute difference between one in every ten duplicates is plotted on the Shewhart plot which is appropriate for that analysis and sample value. These results are kept in the TOC Quality Control notebook.
- 4. The volume of sample is adjusted for the sample analyzed to produce an organic carbon content in the range of optimum function of the instrument.
- 5. Every twentieth sample is spiked and recoveries are determined.
 These results are kept in the TOC Quality Control notebook.
- 6. Every week a seven point standard curve is run by spiking reagent water with seven different concentrations of organic carbon. This curve is kept in the TOC Quality Control notebook.
- 7. Each week a three point standard curve is run by spiking reagent water with three different concentrations of potassium hydrogen phthalate. This curve is kept in the TOC quality control notebook.

- 8.. Each day one standard is run in duplicate. The average of this standard must be within 10 percent of the value for that standard on the weekly standard curve or a new standard curve must be run. These results are kept in the TOC Quality Control notebook.
- 9.. Any maintenance to the instrument is noted in the TOC Quality Control notebook.
- 10. When each new TOC stock standard is prepared, the old standard and the new one are analyzed in triplicate. If the difference between them is greater than 10 percent another standard is prepared. The results of these analyses are kept in the OC Quality Control notebook.

G. Organic Analyses Quality Control Reports

SOLO PERSONAL LIBERTARIA DESCRIPTA DESCRIPTA DESCRIPTA DESCRIPTA DESCRIPTA DESCRIPTA PROGRAMA DESCRIPTA DE CONTRA DE

- 1. After manual review of the data, the analyst submits a report to the lab clerk.
- 2. After reviewing each report the lab clerk submits the report for typing.
- 3. The lab clerk proofreads the typed report, then returns the original report and the typed copy to the analyst.
- 4. The analyst proofreads the report and signs it when satisfied that the results reported are valid.
- 5. The chromatograms and worksheets, if needed, are clipped to the final report and submitted to the Organics Quality Control Manager for final review. As a final check, the values reported are compared to the typical concentration range expected for a particular water supply.
- 6. If the report is approved by the QC Manager the report is signed by him (or her). If not approved the report is sent back to the analyst for review or reanalysis of the sample.
- 7. If approved by the QC Manager, a copy is made of the report and the original sent to the client.
- 8. If possible, the remaining portion of the water sample is stored for one month at 4°C should a repeat of the analysis be necessary.

XII. QUALITY CONTROL - MICROBIOLOGY - EEWTP

A. Sampling

contract to the contract of the contract statement of the contract of the cont

Prior to collecting samples the collector should read <u>Standard Methods</u>. The precautions, summarized below, are mentioned in the text; their purpose is to maintain the integrity of the samples.

- 1. Prevent contamination of the sample bottle by collector.
- 2. Prevent contamination from other sources not representative of sample, e.g., rust from sampling tap and sediment from stream bed.
- 3. Do not rinse the sample bottle with the sample. The bottle contains sodium thiosulfate preservative.
- 4. Leave some air in the container and cap tightly.
- 5. Identify and describe the samples as completely and accurately as possible. This information will aid the analyst in determining the dilutions to inoculate. The following information is to be provided:
 - a. Sampler
 - b. Date and time taken
 - c. Sample number (sampler's own ID notation)
 - d. Remarks (description of sample)
- 6. Samples transported to the laboratory from off-site locations should be packed in ice to maintain a temperature of 4 to 6°C.

B. Acceptance of Samples

Upon arrival at the laboratory, the samples are to be inspected for sample integrity. The samples are logged into the "Sample Log" notebook. A "Bacteriological Work Sheet" is to be filled out for each sample. The time when the samples are inoculated is to be recorded on these worksheets, e.g., 10:15 a.m. This time will indicate when the 24 and 48 hour readings are to be made. All samples are analyzed within four hours of collection. An aliquot of each sample is analyzed to assure the absence of a chlorine residual. Any sample with a residual is discarded.

C. References

- 1. The basic methods reference is Standard Methods for the Examination of Water and Wastewater, 13th Edition. Use of the procedures in this edition of Standard Methods is mandated under the National Interim Primary Drinking Water Regulations.
- 2. EPA's Microbiological Methods for Monitoring the Environment -Water and Wastes write-up is useful.

D. MPN Analyses

- 1. Five tubes per dilution are to be used for all work. No Exceptions. For drinking water samples for which the analyst has no prior history, three dilutions (10 ml, 1.0 ml, and 0.1 ml) should be inoculated.
- 2. Three tubes per dilution may be used for pilot plant work. This option is to be determined on a case-by-case basis by a senior staff member and the project engineer.
- 3. The completed MPN test is performed on all positive confirmed BGB tubes from sites representing finished waters.

E. Standard Plate Count

- 1. Duplicate plates per dilution are to be routinely used. A senior staff member is to be consulted for the dilutions to be examined.
- 2. Plates having 30 to 300 colonies are to be included in determining the number of bacteria per milliliter. The plates are to be counted on the Quebec Colony Counter.
- 3. If plates show no colonies, report the count as less than one (1) times the corresponding lowest dilution. If the plate shows numbers of colonies exceeding 300, do not report as "too numerous to count" (TNTC) but utilize the procedure in the 13th edition of Standard Methods to estimate the count.
- 4. Standard plate counts are to be recorded with not more than two significant figures, thus avoiding false precision and accuracy.

F. Steam Sterilization

- 1. On the front of the autoclave are a set of instructions for the operation of the Market Forge autoclave. The lab assistant should note that the timed cycle does not start until the pressure reading is 15 psi and temperature is 121°C. The material, time, pressure, and temperature are to be recorded in the Sterilization Log book.
- Sterilization indicators are available and one <u>must</u> be used during each cycle, these include: Kilit^R Ampules, heat sensitive tape, steam clocks, or discs. If problems exist, indicated by a failure of one of the various sterilization indicators, consult with a senior staff member.
- 3. All plastic and glassware to be autoclaved are to have indicating autoclave tape placed on it.
- 4. Borosilicate test tubes filled with media are autoclaved in autoclaveable plastic test tube racks.
- 5. Media does not stay in the autoclave longer than 45 minutes.
- 6. Sterilization times are determined from the time the temperature reaches 121°C until the cycle is complete:

- a. Media according to manufacturer's instructions
- b. Sample bottles 30 minutes, 15 psi
- c. Filter units 30 minutes, 15 psi
- d. Dilution water less than 500 ml 30 minutes, 15 psi
- e. Dilution water 500 to 1000 ml 45 minutes, 15 psi

G. Dry Heat Sterilization

- 1. Wooden transfer sticks and most glassware are sterilized by dry heat for two hours at 170 to 180°C.
- 2. Sterilization records are kept in a log similar to the one used for autoclaving.

H. Media Preparation and Laboratory Pure Water

- Only Milli-Q or demineralized water can be used for the preparation of media at the EEWTP.
- 2. Dehydrated media and pH buffers must be dated upon receipt and when initially opened.
- 3. Rehydrated media is stored at room temperature in the dark or at 4°C with the provision that refrigerated media will be incubated overnight at 35°C prior to inoculation. All media should be labeled with the type of medium, date and the person who made it up.
- 4. Media pH is recorded in the media pH log. This log also documents meter calibration, date of preparation and type of media prepared.
- 5. The water suitability test as described in Standard Methods, 14th Edition, is conducted once per year on laboratory pure water (Milli-Q).
- 6. Laboratory pure water is analyzed monthly (minimum) for conductance, pH, chlorine residual and standard plate count.
- 7. Laboratory pure water is analyzed annually (minimum) for trace metals (Pd, Cd, Cr, Cu, Ni and Zn).
- 8. Inhibitory residue test is conducted annually according to Standard Methods, 14th Edition.



TO VEVERSE VICTORIA AN PERSONAL RECEIPER MAINTAIN CHECKEN FESSOOF MAINTAIN PROCEED FOR

I. Sample Bottle Preparation

- 1. The normal washing procedure is to be used.
 - a. Alconox or Micro
 - b. Rinse with tap water four times
 - c. Rinse with deionized water four times
- 2. After the bottles have dried, 0.1 ml of a 10 percent sodium thiosulfate solution is added to each bottle. Autoclave tape is to be used on the cap. The bottles are then loosely capped and steam sterilized. After the bottles have cooled to room temperature, the caps are tightened and placed in the drawer labeled "Sample Bottles." If there are any questions about sterility of bottles, they should be reautoclaved.
- 3. Sample bottle sterility checks are performed with each newly sterilized batch.

J. Incubator and Water Bath Operations

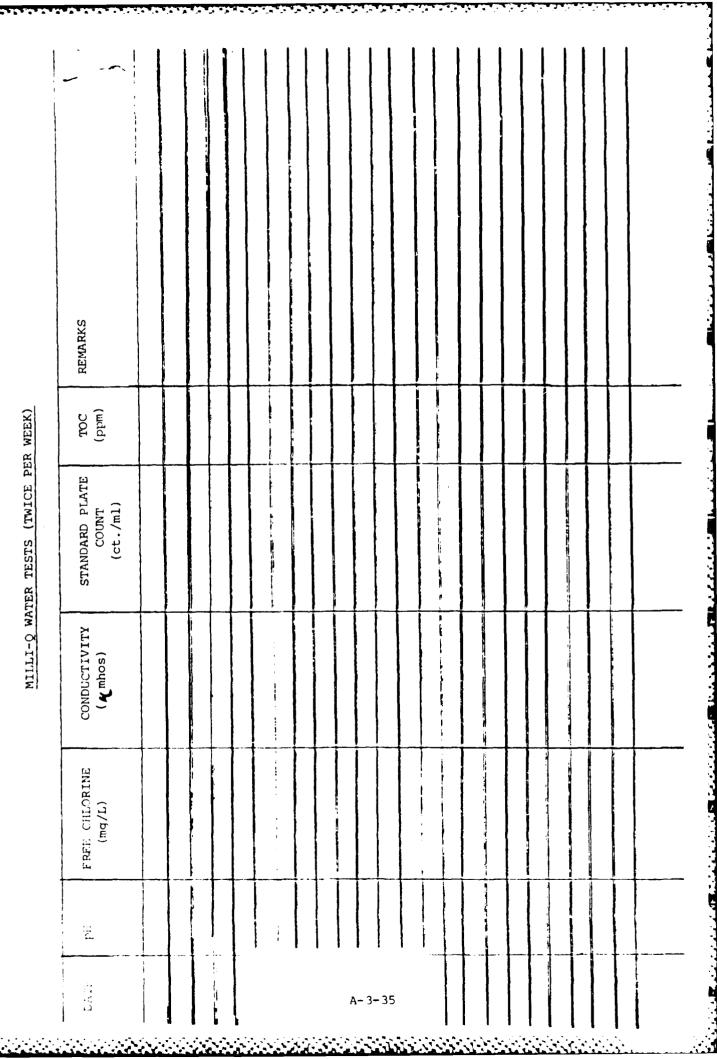
- 1. On a daily basis when bacteriological samples are incubated in an incubator or water bath, the temperature is to be recorded each morning in the appropriate log. Adjustments are made when necessary.
- 2. Two thermometers are placed in the incubator on different shelves to monitor temperature uniformity. Each thermometer is immersed in water.
- Thermometers are calibrated with an NBS-calibrated thermometer on a semi-annual basis.
- 4. Tubes and plates are placed in the incubator so that there is adequate air circulation.

K. Quality Assurance

- 1. Positive and negative controls for MPN must be used each time the analysis is performed. Positive controls utilize a known coliform such as <u>E. coli</u> or <u>E. aerogenes</u>. Negative controls should be a sterile phosphate buffer.
- 2. Written records of quality control on media, materials, and equipment must be maintained and available for inspection. This information is to be logged into the appropriate log sheets.
- 3. Media supplies must be dated upon receipt and when initially opened. A written record must be maintained on media. When receiving new media, the date, the type of media, source of media, and initials of the person logging in the media are to be recorded in the Media Log book.

- 4. Operating temperatures of incubators, waterbaths, hot air ovens, and refrigerators must be checked daily, logged in the appropriate book and adjusted if needed.
- 5. Autoclave temperatures and pressures must be logged in the autoclave log for each cycle of use. Sterilization indicators must be used each time the autoclave is operated.
- 6. Before use in an analysis each pH meter must be standardized. This is recorded in the media pH log.
- 7. Balances are calibrated and certified with NBS Class S weights on a semi-annual basis.





*Thermometer must be in water.

	REMARKS (,													
	THERMOMETER II bottom														
(DAILY)	THERMOMETER I top														
	DATE TH														
* INCUBATOR TEMPERATURE	REMARKS														
	THERMOME FER II bottom		,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,			-									
·	THERMOMETER I top														
	DATE						A	- 3-	36	ı				Ì	

SANTON TRANSPART BRACKERS TO SERVICE

MICRO STERILIZATION LOG: BLUE-M

BLUE-M OVEN

	MAXIMUM TEMPERATURE	OBTAINED									The state of the s						the second of the second secon		
	OVEN;	TOTAL TIME ELAPSED								:					to be a second s				
	TIME IN	TIME .							1		;								
	LENGTH OF	TIME									1		A de la companya de l				1		
	ITEMS STERILIZED	(SPECIFY ITEMS)										and the second s		 to temporary to the second second second second second	:	 			
 	DATE				!	w. .w.c.	i	, ,	A-3	3-37 - 1-1	7		ار د د د د						

APPROPRIATE AUTOCIAVE TIMES:

THE PROPERTY OF THE PROPERTY O

15 MINUT 30 MINUT 30 MINUT 30 MINUT PLASTIC SAMPLE BOTTLES: 3
MILLIPORE FILTER UNITS: 3
DILUTION MILK BOTTLES: 3
(PEPTONE DILUTION WATER)

> AUTOCLAVE MICRO STERILIZATION LOG:

MAXIMUM PRESSURE OBTAINED MAXIMUM TEMPERATURE OBTAINED TOTAL TIME ELAPSED LENGTH OF TIME IN AUTOCLAVE: TIME TIME filter units, etc.) (Media, glassware, ITEMS STERILIZED DATE

A-3-38



PH LOG FOR MEDIA

- -	-	(3	٤.																
REMARKS		:			:					:										 1		
G BOTH BUFFERS:	рн 10	1	! !	 :			 -		,				,		_		:	!				
RECORD PH USING	pH 4								:	1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	a spirit de sera labor de aprime e application de laboration de la companya de la					i i					The same of the sa	
CALIBRATION AT PH7	(Reading)					1						•										
SPECIFY TYPE OF	MEDIA (BGB,LIB,EC, EMB,SPCA,etc.)																			and the state of t		
DATE PREPARED							A-	- 3-	39													

		DATE			HOURS	SAD NO		-
COLLE	CTED					ANALYST		
EXAMI	NE D							
		M	PN					
	PRESU	MPTIVE		RMED	EC	Standard	Veri	fied _
ml D⊢L	LT	В	BG	L B	44 5·C	_Plate Count_	Ţ.	гв
	35	,C	35	.с			35	·c
	24 hrs	48 hrs	24 hrs	48 hrs	24 hrs	48 hrs	24 hrs	48 hrs
]				
10								
					<u> </u>	J		
				<u> </u>	<u> </u>			ļ
			!		<u> </u>	<u>-</u>		<u> </u>
10			 			┤ ├─── ┦	<u> </u>	
,0			 	 	 -	1	-	
	 				}	1		
						₹ ├── 		├
			 	 		1		
10			† — —	<u> </u>		†		
			j			11		
_								†
								†
10]		
	<u> </u>		ļ					
	!		ļ		<u> </u>	┥┫		<u> </u>
			ļ	 	<u> </u>			
10					I	 	<u> </u>	
10	<u> </u>	 	 	 	 	┥ ──┪	<u> </u>	
		 	 	 	-	1	<u> </u>	 -
		L	<u> </u>				L	ــــــــــــــــــــــــــــــــــــــ
			* * *					
CAL CO	DLIFORM M	PN		PER 10	0 ML			
MARKS	<u> </u>					····		_
								_

A- 2- 40

Chemical Reagent

Name:						Reald:	
Lot /St	RNO:		•	Manu	Sacturer:		
Quanti	ty Record:		_ Size	Recuid:			معادت
EXD Da	•		· · · · · · · · · · · · · · · · · · ·				
Batch	Make U	0:					
Date	Date Solution Made	Amt.	Volume Made (MIS)	Date Solution Expires			
		(01	(1115)				
	<u> </u>						
		l I					
				-			
		<u> </u>					
İ	ı						
1							
	· t	Ì	1				

ATTACHMENT 1
SAMPLE COLLECTION

SAMPLING CONTAINERS AND PRESERVATIVES

All samples (except Metals) are required to be refrigerated at 4°C unless otherwise indicated. Sodium thiosulfate should only be used in the presence of residual chlorine. No attempt should be made to use the same sample for chemical, bacteriological and microscopic examination because the methods of collection and handling are quite different.

DETERMINATION	CONTAINER	ROUTINE SAMPLE SIZE	PRESERVATIVE OR NEUTRALIZER (per EPA)	MAX. UM HOLDING PERIOD (per EPA
Acidity	Plastic or pyrex bottle, Tightly sealed, no bubbles	125 ml	None	14 days
Acids	Amber glass, teflon lined cap, muffled	2 liter	0.008% sodium thiosulfate	14 days
Actinomycetes	Plastic (sterile)	125 ml	0.008% sodium thiosulfate	24 hours
Alkalinity	Plastic, Tightly sealed, no bubbles	125 ml	None	14 days
Algae	Plastic (sterile)	125 ml	.008% sodium thiosulfate	24 hours
Algae Count	Plastic (sterile)	l liter	.008% sodium thiosulfate	24 hours
Aluminum	Plastic, HNO3 rinse	60 ml	HNO ₃ to pH<2	6 months
Anions	Plastic	500 ml	None	14 days
Antimony	Plastic, HNO3 rinse	60 ml	HNO ₃ to pH < 2	6 months
Arsenic	Plastic, HNO3 rinse	60 ml	HNO ₃ to pH < 2	6 months
Asbestos	Plastic (sonicated)	l liter	None	2 days
Barium	Plastic, HNO3 rinse	60 ml	HNO3 to pH <2	6 months
Base-Neutrals (BN)	Amber glass, teflon lined cap, muffled	2 liter	.008% sodium thiosulfate	14 days
Base-Neutral- Acids (BNA)	Amber glass, teflon lined cap, muffled	2 liter	.008% sodium thiosulfate	14 days
Beryllium	Plastic, HNO3 rinse	60 ml	HNO ₃ to pH<2	6 months
Biological Oxygen Demand (BOD)	Plastic	500 ml	Freeze	2 days
Boron	Plastic, HNO ₃ rinse	60 ml	HNO ₃ to pH<2	6 months
Bromide	Plastic	60 ml	None	28 days
Cadmium	Plastic, HNO ₃ rinse	60 ml	HNO ₃ to pH < 2	6 months
Calcium	Plastic, HNO3 rinse	60 ml	HNO ₃ to pH < 2	6 months
Carbon Dioxide (Free)	Plastic, tightly sealed, completely filled, allow to overflow.	500 ml	None	Titrate at site or measure pH at site and do alkalinity at lab.
Cations	Plastic, HNO3 rinse	500 ml	HNO ₃ to pH <2	6 months
Chemical Oxygen Demand (COD)	Plastic	125 ml	H ₂ SO ₄ to pH<2	2 d a ys
Chloride	Plastic	60 ml	None	28 days



SECON SECURITY WAS AND A CONTRACT OF THE PROPERTY OF THE PROPE

SAMPLING CONTAINERS AND PRESERVATIVES (CONTINUED)

All samples (except Metals) are required to be refrigerated at 4°C unless otherwise indicated. Sodium thiosulfate should only be used in the presence of residual chlorine.

DETERMINATION	CONTAINER	ROUTINE SAMPLE SIZE	PRESERVATIVE I OR NEUTRALIZER (per EPA)	MAXIMUM HOLDING PERIOD (per EPA)
Chlorine-Residual	Amber glass, teflon septa, muffled	250 ml	Determine on site, Keep in the dark	2 hrs.
Chlorine-Demand	Plastic	1 liter	Freeze	3 days
Chromium- Hexavalent	Plastic, HNO ₃ rinse	60 ml	None	2 days
Chromium-Total	Plastic, HNO ₃ rinse	60 ml	HNO ₃ to pH<2	6 months
Closed Loop Stripping (CLS)	Amber glass, teflon lined cap, muffled	1 liter	50 ppm mercuric chloride (HgC	l ₂) 21 days
Cobalt	Plastic, HNO ₃ rinse	60 ml	HNO ₃ to pH<2	6 months
Coliform (MPN)	Plastic (sterile)	125 ml	0.008% sodium thiosulfate	30 hours
Color	Amber glass	125 ml	None	2 days
Copper	Plastic, HNO3 rinse	60 ml	HNO ₃ to pH<2	6 months
Cyanide	Plastic	60 ml	NaOH to pH>12 0.008% sodium thiosulfate	14 days
Detergents		See Surfactants		
Fecal Coliform (MPN)	Plastic (sterile)	125 ml	0.008% sodium thiosulfate	30 hours
Fecal Streptococcus(MPN)	Plastic (sterile)	125 ml	0.008% sodium thiosulfate	30 hours
Fluoride	Plastic	60 ml	None	28 days
General Mineral Analysis	Plastic	2-500 ml	l bottle with HNO ₃ to pH<2 l bottle with no preservative	14 days
Geosmin/MIB.		See MIB/Geosmin		
Grease		See Oil	and Grease	
Hardness	Plastic, HNO3 rinse	125 ml	HNO ₃ to pH<2	6 months
Herbicide	Amber glass, teflon lined cap, muffled	2 liter	0.008% sodium thiosulfate	14 days
Iodide	Plastic	60 ml	None	28 days
Iron (Total)	Plastic, HNO3 rinse	60 ml	HNO ₃ to pH<2	6 months
Lead	Plastic, HNO ₃ rinse	60 ml	HNO ₃ to pH<2	6 months
Lithium	Plastic, HNO3 rinse	60 ml	HNO3 to ph<2	6 months

SAMPLING CONTAINERS AND PRESERVATIVES (CONTINUED)

All samples (except Metals) are required to be refrigerated at 4°C unless otherwise indicated. Sodium thiosulfate should only be used in the presence of residual chlorine.

DETERMINATION	CONTAINER	ROUTINE SAMPLE SIZE	PRESERVATIVE OR NEUTRALIZER (per EPA)	MAXIMUM HOLDING PERIOD (per EPA)
Magnesium	Plastic, HNO3 rinse	60 ml	HNO3 to pH <2	6 months
Manganese	Plastic, HNO ₃ rinse	60 ml	HNO3 to pH < 2	6 months
MBAS (Methylene Blue Active Substances)		See Surfactants		
Mercury	Plastic, HNO ₃ rinse	250 mı	HNO3 to pH < 2	28 days
Metals Total (in general mineral)	Plastic, HNO3 rinse	500 ml	HNO3 to pH<2	e months
MIB/Geosmin	Amber glass. eflon septa, muffled	250 ml	50 ppm mercuric chloride (HgCl ₂)	21 days
Molybdenum	Plastic, HNO ₃ rinse	60 ml	HNO3 to pH <2	6 months
Nickel	Plastic, HNO3 rinse	60 ml	HNO3 to pH < 2	6 months
Nitrogen-Ammonia	Plastic	125 ml	H ₂ SO ₄ to pH<2	28 days
Nitrogen-Nitrate	Plastic	125 ml	None	2 days (14 days for drinking H ₂ O)
Nitrogen-Nitrite	Plastic	125 ml	None	2 days
Nitrogen-Organic	Plastic	125 ml	H ₂ SO ₄ to pH<2	28 days
Nitrogen-Kjeldahl Total	Plastic	125 ml	H ₂ SO ₄ to pH<2	28 days
Nutrients	Plastic	125 ml	H ₂ SO ₄ to pH<2	28 days
Organic Carbon	Organic Carbon See Total Organic Carbon			
Odor	Odor-free glass, no air, completely filled	500 ml	None	24 hours
Oil and Grease	Widemouth glass	1 liter	H ₂ SO ₄ to pH<2 Sludge sample; 1 ml Conc H ₂ SO ₄ /80 gm of sludge	28 days
Oxygen, Dissolved	Glass, fill to neck	250 ml	Determine on site	1 hour
Pesticide	Amber glass, teflon lined cap, muffled	2 liter	0.008% sodium thiosulfate	14 days
Pesticide/PCB	Amber glass, teflon lined cap, muffled	2 liter	0.008% sodium thiosulfate	14 days
рH	Plastic	250 ml	None	2 hours
Phenol	Glass	250 ml	H2SO4 to pH<2	26 days

AS DOWN DIVING BONN RESERVE BONN ABOUT BONN RESERVE

SAMPLING CONTAINERS AND PRESERVATIVES (CONTINUED)

All samples (except Metals) are required to be refrigerated at 4°C unless otherwise indicated. Sodium thiosulfate should only be used in the presence of residual chlorine.

DETERMINATION	CONTAINER	ROUTINE SAMPLE SIZE	PRESERVATIVE OR NEUTRALIZER (per EPA)	MAXIMUM HOLDING PERIOD (per EPA)
Phosphate (ortho)	Plastic, HCl rinse	125 ml	H2SO4 to pH<2	2 days
Phosphate (Total	Plastic, HCl rinse	125 ml	H ₂ SO ₄ to pH<2	28 days
Fotassium	Plastic, HNO ₃ rinse	60 ml	HNO3 to pH<2	6 months
Foiychiorobiphenyl (PCB)	Amber glass, teflon lined cap, muffled	2 liter	0.008% sodium thiosulfate	14 days
Polynuclear- aromatic Hydro- carbons (PNA)	Amber glass, teflon lined cap, muffled	2 liter	0.008% sodium thiosulfate	14 days
Purgeables		See VOA		
Radiation	Plastic (2 bottles)	1 liter 125 ml	l bottle with HNO _{3 to pH<2} l bottle with no preservative	6 months
Radium	Plastic	1 liter	HNO ₃ to pH<2	6 months
Residue	See Solids			
Selenium	Plastic, HNO ₃ rinse	60 ml	HNO3 to pH<2	6 months
Settleable Solids	Plastic	1 liter	None	7 days
Silica	Plastic	60 ml	None	28 days
Silver	Plastic, HNO ₃ rinse	60 ml	HNO ₃ to pH<2	30 days
Sodium	Plastic, HNO ₃ rinse	60 ml	HNO3 to pH<2	6 months
Solids	Plastic	500 ml	None	7 days
Specific Con- ductance	Plastic	250 ml	None	28 days
Strontium 90	Plastic	1 liter	HNO ₃ to pH<2	6 months
Sulfate	Plastic	60 ml	None	28 days
Sulfite	Plastic, tightly sealed, no bubbles, fill to neck	60 ml	None	48 hours
Sulfide (Total)	Plastic (no bubbles) fill to neck	60 ml	Field analyses or 4 drops of 2N acetate/60ml	28 days
Surfactants	Plastic	250 ml	None	48 hours
Taste	Glass	500 ml	None	48 hours
Tetrachloroethene (PCE-	Amber glass, teflon septa, muffled	125 ml	0.008% sodium thiosulfate	14 days

CONTROL OF STREET STREE

SAMPLING CONTAINERS AND PRESERVATIVES (CONTINUE)

All samples (except Metals) are required to be refrigerated at 4°C unless otherwise indicated. Sodium thiosulfate should only be used in the presence of residual chlorine.

DETERMINATION	CONTAINER	ROUTINE SAMPLE SIZE	PRESERVA TIVE OF NEUTR ALIZE R (per EPA)	MAXIMUM HOLDING PELIOD (per EFA
Thallium	Plastic, HNO3 rinse	60 ml	HNO3 to pH <2	6 months
Thiosulfate	Plastic. (no bubbles)	60 ml	None	48 hours
Tin	Plastic, HNO3 rinse	60 ml	HNO ₃ to pH<2	6 months
Titanium	Plastic, HNO3 rinse	60 ml	HNO ₃ to pH<2	6 months
Total Dissolved Solids (TDS)	Plastic	500 ml	None	14 days
Total Organic Carbon (TOC)	Amber glass, teflon septa, muffled	125 ml	H ₂ SO ₄ to pH<2	28 days
Total Organic Halogens (TOX)	Amber glass, teflon septa, muffled	250 ml	H ₂ SO ₄ to pH<2 and 2 drops of 0.1M sodium sulfite	14 days
Total Plate Count (TPC)	Plastic (sterile)	125-500 ml	0.008% sodium thiosulfate	30 hrs
Total Potential Tribalomethane (TPTHM)	Amber glass, teflon septa, muffled	125 m!	None	14 days
Total Potential Trihalomethane Curve (TPTHM)	Amber glass, teflon lined cap, muffled	2 liter	None	14 days
Total Suspended Solids (TSS)	Plastic	500 ml	None	7 days
Trace Metals	Plastic, HNO3 rinse	500 ml	HNO ₃ to pH<2	e months
Trichloroethene (TCE)	Amber glass, teflon septa, muffled	125 ml	0.008% sodium thiosulfate	14 days
Trihalomethane (THM)	Amber glass, teflon septa, muffled	125 ml	0.008% sodium thiosulfate	14 days
Turbidity	Plastic	125 ml	Store in dark	48 hours
Vanadium	Plastic, HNO3 rinse	60 ml	HNO ₃ to pH<2	6 months
Volatile Acids	Plastic	125 ml	None	7 days
Volatile Organic Acids (VOA)	Amber glass, teflon septa, muffled	250 ml	0.008% sodium thiosulfate	14 days
Volatile Suspended Solids (VSS)	Plastic	500 ml	None	7 days
Zinc	Plastic, HNO ₃ rinse	60 ml	HNO3 to pH-2	5 menths

ATTACHMENT 2

SHEWART CONTROL CHARTS

Shewhart Quality Control Charts

THE STANSON SANDONN THERED

Dr. Walter A. Shewhart of Bell Telephone Laboratories developed the basic theory of control charts in the 1920's. His book on statistical quality control (1) grew out of this original work. Since then, industrial acceptance of these control chart concepts and other statistical techniques have refined and quantitated the quest for quality in manufacturing. Although originally developed for control of production processes when large numbers of articles were being manufactured and inspected on an essentially continuous basis, these same concepts have been readily adapted to laboratory operations where the analyst produces comparatively fewer results on an intermittent basis.

As in the CuSum approach, precision control charts are prepared from data resulting from duplicate sample analyses and accuracy control charts from duplicate spiked standards or samples. Once the control charts are constructed, however, data are plotted as individual values rather than cumulative sums.

Certain constants (factors) are also involved in the preparation of Shewhart Charts. Depending upon how the data are grouped, what the size of each grouping is, and what control limit formulation is being calculated, Table 6-3 will serve as a basic reference point:

Table 6-3

FACTORS FOR COMPUTING CONTROL CHART LINES (5, 6)

Observations in Subgroup (7)	Factor A ₂	Factor D ₄
2	1.88	3.27
3	1.02	2.58
4	0.73	2.28
5	0.58	2.12
6	0.48	2.00
7	0.42	1.92
8	0.37	1.86

Inherent in the Shewhart approach is recognition of the basic assumption that variations exist in every method. That is, no procedure is so perfect, so unaffected by its environment, that it will always give exactly the same assay value or product. Where such situations seem to exist, either the device used to measure the process is not sensitive enough or the person making the measurements is not performing properly. For our purposes, the recorded difference between paired samples should never be less than one-half the minimum detectable limit of the parameter under consideration. In the following outlines for preparing precision and accuracy control charts, nitrate data were used to develop the examples. Therefore the minimum values shown are 0.05 (one-half the observed minimum detectable limit of 0.1 mg/1 as N).

Precision Control Charts

These charts are developed by collecting data for many samples, a minimum of 15 to 20, run in duplicate under assumed controlled conditions. Once these data have been generated, preferably over an extended period of laboratory time, the following steps should be followed to construct the control chart:

a. List the range (R) for each set of samples. That is, the absolute value of the difference between each set of duplicate samples.

Note: The following ranges were observed in the nitrate data:

0.1	0.1	0.05
0.05	0.05	0.05
0.05	0.05	0.05
0.05	0 .05	0.05
0.05	0 .30	0 .05
0.05	0.10	0.05
0.05	0.05	0.05
0.05	0 .05	0.05
0.05	0.05	0.05
0 .05	0.05	0.05
0 .05	0.05	0.05
0.05	0.05	

b. Calculate the average range (\overline{R}) by summing the list of R values and dividing by the number of sets of duplicates:

$$\overline{R} = \frac{\Sigma R}{7}$$

$$\overline{R} = \frac{2.15}{35} = 0.06$$

c. Calculate the Upper Control Limit (UCL) on the range according to the formula

$$UCL_R = D_4 \overline{R}$$
,

where D_4 is a constant dependent on the number of units in the subgroup. In this case, since two observations are in the subgroup, $d_4 = 3.27$ (see Table 6-3).

$$UCL_R = 3.27 \ \overline{R} = 0.20$$

d. Calculate the Upper Warning Limit (UWL) on the range according to the formula

$$UWL_R = 2/3 (D_4 \overline{R} - \overline{R}) + \overline{R},$$

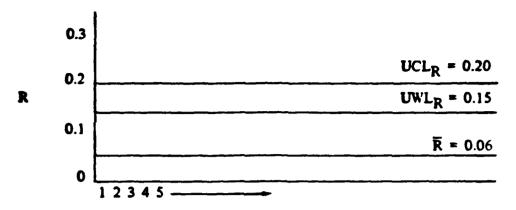
which for duplicate samples reduces to

$$UWL_R = 2.51 \overline{R}$$

$$UWL_R = 0.15$$

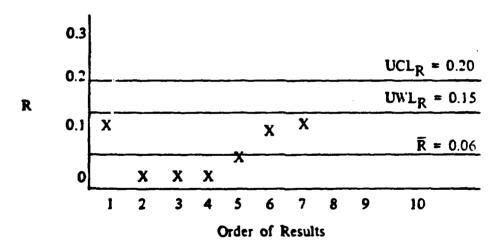
This UWL corresponds to the 95% confidence level.

e. Now graph R, UWLR, and UCLR in the following manner:

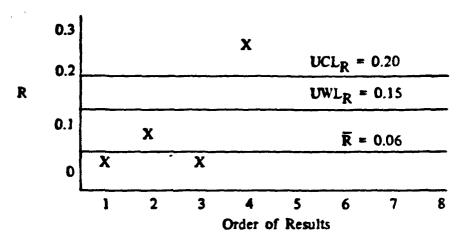


Order of Results (e.g., duplicate sample sets)

- f. The above precision control chart for nitrates is now complete, and can be used to plot R values on subsequent duplicate samples to determine if the system is in control, out of control (plotted R value beyond the UCL), and/or to detect any trends developing within the system.
 - 1. In this example, a trend has developed between duplicate sample sets 4 thru 7. Although the system is not out of control, all variables in the procedure should be checked in an attempt to stop this obvious trend before the UCL is reached.



2. In this example, the system has clearly gone out of control between duplicate samples 3 and 4. At this point, the system can be stopped and all variables in the system checked, or another set of duplicate samples can be run to verify the observed difference. Once the system has been corrected, all samples between set 3 and 4 should be rerun to insure the validity of the data.



Accuracy Control Charts

A STATE OF THE STA

As in the above system, these charts are developed by collecting data for many samples, a minimum of 15 to 20, but on spiked samples (preferably) or standards under assumed controlled conditions. Again, these data should be generated over an extended period of laboratory time, and be representative of normal operating conditions (7). The following steps should be followed to construct accuracy control charts:

a. List the range (R) and the average (X)* of each subgroup of data.

Note: In the following example of nitrate data, subgroups of monthly data involving four observations were used:

$$\overline{X} = \underbrace{\Sigma d \text{ Found - Actual}}_{D}$$

Month	Actual	Found	<u>_R</u>	<u> </u>
Sept.	1.1	1.1		
	1.1	1.1	0	0
	1.1	1.1	•	
	1.1	1.1		
Nov.	1.1	1.1		
	1.1	1.1	0.1	-0 .025
	1.2	1.2		
	1.2	1.1		
Dec.	1.0	1.1		
	1.0	1.0	0.1	+0.025
	1.0	1.0		
	1.0	1.0		
Jan.	1.0	0.9		
	1.0	1.0	0.2	0
	1.0	1.1		
	1.0	1.0		

b. Calculate the average range (\overline{R}) by summing the list of R values and dividing by the number of subgroups:

$$\bar{R} = \frac{\Sigma R}{\eta}$$

$$\overline{R} = \frac{0.4}{4} = 0.10$$

c. Calculate the Upper Control Limit (UCL) on the range according to the formula:

$$UCL_R = D_4 \overline{R}$$
,

where D_4 is a constant dependent on the number of units in the subgroup. In this case, since four observations are in the subgroup, $D_4 = 2.28$ (see Table 6-3).

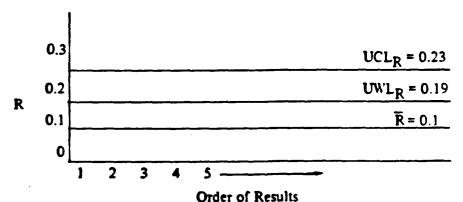
$$UCL_R = 2.28 \bar{R} = 0.23$$

d. Calculate the Upper Warning Limit (UWL) on the range according to the formula:

$$UWL_R = 2/3 (D_A \overline{R} - \overline{R}) + \overline{R}$$

$$UWL_R = 2/3 [2.28 (0.1)-0.1]+0.1 = 0.19$$

e. Now graph \overline{R} , UWL_R, and UCL_R in the following manner:



f. Turning now to the \overline{X} values, calculate the UCL \overline{X} by the formula:

$$UCL\bar{\chi} = A_2\bar{R}$$

where A_2 is a constant dependent on the number of units in the subgroup. In this case, since four observations are in the subgroup, $A_2 = 0.73$ (see Table 6-3).

:
$$UCL\bar{X} = 0.73\bar{R} = 0.07$$

g. Calculate the UWL \overline{X} by the formula:

STATISTICS SOUTHER SECRETARY SOUTHER STATISTICS SOUTHER

UWL
$$\bar{\chi}$$
 = 2/3 A₂(\bar{R})

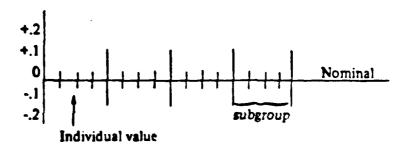
UWL $\bar{\chi}$ = 2/3 [0.73 (0.1)] = 0.05

Note: Lower Warning Limit (LWL $\overline{\chi}$) and Lower Control Limit (LCL $\overline{\chi}$) are simply the negative values of UWL $\overline{\chi}$ and UCL $\overline{\chi}$, respectively.

h. Now graph the standard Nominal Value (set equal to zero), $UWL_{\overline{X}}$, and $UCL_{\overline{X}}$. LWL $_{\overline{X}}$, and LCL $_{\overline{X}}$ in the following manner:

.07	$UCL_{\overline{X}} = 0.07$
.05	$\overline{UWL}\overline{\chi} = 0.05$
.03	
.01	Std Nominal Value
01	1 2 3 4 5 order of subgroups
03	
05	$LWL\overline{\chi} = 0.05$
07	$LCL_{\overline{X}} = 0.07$

i. In order to detail any trends forming within each subgroup, individual differences may be plotted by preparing the following graph:



j. Thus, as in the precision control charts, once all of the above accuracy control charts have been constructed, all future data can be plotted on each set of duplicate spiked samples or standards, to determine if the system is in control, out of control, and/or to detect any trends developing within the system.

References

- 1. Shewhart, W. A., Economic Control of Quality of Manufactured Product, 1931.
- 2. Anon., "Statistical Method-Evaluation and Quality Control for the Laboratory", DHEW Training Course Manual in Computational Analysis, August 1968.
- 3. Anon., "Laboratory Quality Control Manual", Federal Water Pollution Control Administration, Robert S. Kerr Water Research Center, 1969.
- 4. Griffin, D. F., "Systems Control by Cumulative Sum Method", Amer. J. Med. Tech., 34, 644 (1968).
- 5. Duncan, A. J., Quality Control and Industrial Statistics, 3rd Ed., R. D. Irwin, Inc., Homewood, Ill., Chap. 18 (1965).
- 6. ASTM Special Technical Publication No. 15-C, "Manual on Quality Control of Materials", pp. 59-64, January 1951.
- 7. Grant, E. G., Statistical Quality Control, 3rd Ed., McGraw-Hill, New York (1964).

SECTION 4

COMPARATIVE ODOR TESTING

Results of the comparison odor testing performed by the off-site laboratory (JMM, Pasadena) and WAD (U.S. Army Corps of Engineers, Washington Aqueduct Division) on RWQTP duplicates taken from carbon column effluent and finished water sites are summarized below. The results seem to indicate that the olfactory sensitivity of the off-site laboratory panel is greater than WAD panel and this sensitivity applies to a variety of odor types.

CONTROL SYSTEMS CONTROLS PROMOTE CONTROLS

KICKER STREET WESTERN LANDS ABOUT

The differences in odor numbers reported by the two labs may also be due to the distance at which the odor flask is held from the nose while sniffing. Members of the off-site laboratory panel placed their noses directly into the flask rather than holding the flask at mouth level. The odor procedure in Standard Methods does not address the issue of what distance the flask should be held from the nose and so there is no uniform protocol for individual labs to follow. If the WAD panel conducted their odor testing by holding the flask slightly below the nose then it could explain the reason why their reported odor numbers on the WAD duplicate samples are lower than those of the off-site lab.

		ımn Effluent : 390)	EEWTP Finished Water (Site 471)			
Sampling Date	JMM (TON)	WAD (TON)	JMM (TON)	WAD (TON)		
2/27/83	4	<1	40	2		
3/1/83	17	<1	40	2		
3/6/83	8	<1	67	2		
3/8/83	<1	1	40	4		

Note: TON values can be converted to % samples in dilution by taking the reciprocal of the TON value and multiplying by 100.

The odor descriptions given by the off-site laboratory panel for the samples are as follows:

<u>Date</u>	<u>Site 390</u>	<u>Site 471</u>
2/27	Earthy	Sweet, Rancid
3/1	River, Garbage	Rotten Eggs
3/6	Sweet	Chlorine
3/8	Musty	Chlorine

REFERENCES

- Bynum M., S. Tyree, and W. Weiser, "Effect of Major Ions on the Determination of Trace Ions by Ion Chromatography," Anal. Chem., 53, 1935 (1981).
- Chang, S.L., Bert, G., Busch, K.A., Stevenson, R.E., Clark, N.A. and Kabler, P.W., "Application of the Most Probable Number Method for Estimating Concentrations of Animal Viruses by the Tissue Culture Technique," Virology 6:27-42. (1958).
- Jenke, D., "Anion Peak Migration in Ion Chromatography," Anal. Chem, 53, 1536 (1981).
- Katzenelson, E., Fattal, B. and Hostoverskty, T., "Organic Flocculation; An Efficient Second-Stage Concentration Method for the Detection of Viruses in Tapwater," Appl. Environ. Microbiol. 32:638 (1976).
- Sichman, M. and Pyen, G. S., "Determination of Selected Anions in Waste by Ion Chromatrograph," U.S. Water Res. Invest., 79-101, 30p (1979).
- Small, H, Stevens, T., and Bauman, W., "Novel Ion Exchange Chromatographic Method Using Conductimetric Detection," Anal. Chem., 47:1801 (1975).

LEGICAL LEGICALE RESIDENT LEGISLES SESSONS SESSONS SESSONS L'ARRESTA SESSONS L'ARRESTA SESSONS L'ARRESTA (DES)

APPENDIX B DATA MANAGEMENT

CONSTRUCTION CONTRACTOR SECURITION CONTRACTOR CONTRACTOR

SECTION 1

DATA ANALYSIS TECHNIQUES

SELECTION OF A DISTRIBUTION MODEL

ALTERNATIVE MODELS

When the concentration of a water quality parameter at a given sampling location is treated as a random variable it can take on any value over some range subject to the influence of stochastic events. The concentration can be characterized by a probability distribution function, f(x). Such a distribution function (also known as a "probability density function" or p.d.f.) describes the probability that the variable will fall between two values separated by an infinitesimal difference in the continuum of possible concentrations. Another way to present the distribution is the cumulative probability distribution function or c.d.f. If F(x) is the c.d.f. of a concentration, then the probability that the value of the concentration lies between zero and x is F(x). F(x) is the integral of the probability density function, f(x), from zero to the value. Note that in the case of concentrations of water quality parameters, the probability of a negative value for concentration is zero since this is not possible, thus, zero is taken as the lower limit of integration.

There is a physical basis for a number of well-known distribution models in the statistical literature. Some of these are described below:

1. The normal, or Gaussian model. This is a familiar classical distribution for which the p.d.f. is the "bell-shaped curve." The shape of the distribution is a symmetric curve which drops off to zero in both directions from its maximum at the center, or mean. The normal distribution has very widespread application to physical phenomena because of the Central Limit Theorem, which is a mathematical observation that physical distributions of random variables become normal in appearance.

The Central Limit Theorem states that the distribution of the sum of independent random variables will approach a normal distribution, given certain conditions. Therefore, the normal distribution can serve as an appropriate description of variables which result from the aggregation of small, independent stochastic events, the effects of which are additive. An example would be the contribution of many microscopic deviations contributing to the mass of a manufactured product in an additive way.

The normal distribution is described by two parameters, the population mean (1) and variance (σ^2). Alternatively, the mean and the standard deviation, which is the square root of the population variance, can be used to describe the distribution.



Contract to

CHARLES CHARLES TOURISMENT TOURS OF THE COMMENT OF

The general form of the p.d.f. for the normal distribution is:

$$f(x) = \frac{1}{\sqrt{2\pi\sigma}} \exp(-\frac{1}{2\sigma^2} (x-\mu)^2)$$
 (1)

When a random sample is drawn from a normally distributed population, it is possible to demonstrate that the "best" estimators for the population mean and variance are the estimates

$$\bar{x} = \frac{1}{n} \sum_{i=1}^{n} x_i \tag{2}$$

$$s^{2} = \frac{1}{n-1} \sum_{i=1}^{n} (x_{i} - \bar{x})^{2}$$
 (3)

where $x_1, x_2,...,x_n$ are the values of the samples. Note that \bar{x} is simply the sample mean, and s^2 is the square of the unbiased form of the sample standard deviation. These estimators for the distribution parameters are applicable when (1) data are all greater than the detection limit, and (2) the sample is truly a random sample in which each observation is independent of another. The cases in which these two qualifications fail are described elsewhere.

2. The log-normal model. This is a distribution in which the logarithm of the random variable is normally distributed. The physical basis for this model is analogous to that of the normal model. When the contributions of a set of random variables to an aggregate variable are multiplicative, the contributions of the logarithms of the effects are additive. When the assumptions of the Central Limit Theorem are satisfied, the logarithm of the random variable is normally distributed and the variable itself is lognormally distributed. An example of a phenomenon for which effects are multiplicative is the reproduction of bacteria, where the event of doubling has a multiplicative effect on future populations. This is one reason why the concentration of coliform bacteria in samples tends to be lognormally distributed rather than normally distributed. This is the classic example of the log-normal distribution in water quality (see, for example, Standard Methods).

^{1. &}quot;Best" estimators display a uniform minimum variance from the true value and are unbiased. The concept of a uniformly minimum variance unbiased estimate is further described later in this section.

The log-normal distribution is also a two-parameter model, described by the population geometric mean (μ_g) and population spread factor (S_g) . If the concentration being described is Y, and the random variable X = Log Y is normally distributed, then the geometric mean of Y is equal to the antilogarithm of the arithmetic mean of X, and the spread factor of Y is equal to the antilogarithm of the standard deviation of X.

The "spread factor" is a measure of the dispersion of a log-normally distributed random variable. It is analogous to the standard deviation in normally distributed data, but applies as a multiplicative factor. For example, 68.3 percent of the distribution of Y would fall between μ_g/S_g and μ_g*S_g , while 95.4 percent of the data would fall between μ_g/S_g^2 and $\mu_g*S_g^2$. These correspond to the ranges around the mean of one and two standard deviations for X, the logarithm of Y.

The "best" estimators for the population geometric mean and spread factor of a log-normally distributed variable are:

$$M = \left(\frac{n}{\parallel} y_{i}\right)^{1/n} = 10$$

$$\sum_{i=1}^{n} \log_{10} y_{i}$$

$$\sum_{i=1}^{n} \log_{10} y_{i}$$

$$(4)$$

$$(\frac{1}{n-1} \sum_{i=1}^{n} (\log_{10} y_i - \log_{10} M)^2)^{\frac{1}{2}}$$
(5)

ののかのは、「大きななななない。」というないのでは、このではなから、またいできないというない。

M and S are the sample geometric mean and spread factor, respectively.

- 3. The Gumbel distribution. This represents a case in which the upper tail of the distribution decays in an exponential manner for large values. It has a physical basis in representing a random variable which results from the largest of many random variables which have exponentially decreasing upper tails. It is commonly used in hydrology to describe maximum flood flows. The Gumbel distribution has two parameters.
- 4. The gamma distribution. This two-parameter distribution arises from the sum of independent random variables which are exponentially distributed. It is used in engineering applications because of its convenient shape and skewness.

CANADAS CONTRACTOR DAMAGES INCOME.

5. The Wakeby distribution. This and some other distributions are based on three parameters; the principal advantage is that the increased number of parameters allows a better fit to the data than a two-parameter family. The Wakeby distribution has been proposed for describing runoff.

Of these models for probability distributions, the normal and log-normal models have the widest applicability to physical systems, have an underlying physical

basis, are most commonly used for describing water quality data, and have convenient forms for the parameters which have some physical meaning. These two distributions were selected for further evaluation of applicability in describing water quality data from the EEWTP.

EVALUATION OF MODELS

There are several approaches to evaluating the "goodness-of-fit" of probability distribution models to data in the statistical literature. These include (1) the Kolmogorov-Smirnov test, (2) Pearson's Chi-square test, and (3) the Shapiro-Wilk test, as well as others. None of these tests provide a particularly strong basis for selecting one distribution over another, although once a distribution has been selected they can be effective for demonstrating that a given set of data is inconsistent with a particular distribution.

Kolmogorov-Smirnov Test

The Kolmogorov-Smirnov statistic, D_n , is a measure of the maximum deviation between the value of the "empirical distribution function" of a sample set and the cumulative distribution function of the model being tested. In this case the empirical distribution function, $F_n(x)$, is equal to the number of samples with value less than x, divided by the total number of samples. In a sense, D_n is a measure of the maximum difference along the percentile axis between the actual data on a probability plot and the model being tested. This is not precisely the case because plotting positions used in graphical presentation of the data differ from the values of the steps in the empirical distribution function.

The Kolmogorov-Smirnov statistic has a known probability distribution itself, which can be used in hypothesis testing. A model which is hypothesized to represent the underlying population from which a sample is drawn can be rejected when the measured value of D_n is larger than a critical value. Critical values for the K-S statistic are tabulated in the literature.

In strict mathematical terms, the K-S statistic cannot be used for testing a hypothetical model against data when the parameters of the model are actually estimated from those data. The K-S test applies to normal distributions, for example, only when the population mean and variance are assumed known. In the case when mean and variance (or, geometric mean and spread factor) for log-normal distributions are obtained from the sample values, a modified statistic, D_n^* , based on those values can be employed, but its distribution is different from that of the actual K-S statistic and a different set of critical values must be used. In the case of evaluation of EEWTP data, no prior assumptions about mean and variance are made, so it is appropriate to use a modified statistic, D_n^* . Critical values for the distribution of D_n^* are not available in closed form, but have been estimated from Monte Carlo simulations for the asymptotic case of large sample sizes as described by Stephens, (1974). These critical values have been used here for examining the fit of actual data to normal and log-normal distributions with parameters based upon the data.



Pearson's Chi-Square Test

Pearson's chi-square statistic is a measure of the deviation of observed discretized data from those expected under a given hypothesis. It applies to cases where the number of data falling into a category can be compared to the expected number given by the hypothesis. It can be expressed as the sum of (observed - expected)²/expected over all categories. In the present case, one can divide the distribution of observed measurements of a concentration into a fixed number of concentration ranges. Then "observed" would be the number of points falling within a single concentration range, "expected" would be the number expected in that range given the hypothesized distribution model, and the term would be summed over all concentration ranges to obtain the statistic CS.

In general, it can be shown that performance of the chi-square test is improved when the ranges of concentration are selected to yield equal counts of "expected" values in each range. This was done for the chi-square tests performed during this study, in which the normal and/or log-normal models were used to partition the range of concentrations into groups of ten percentiles per range. Exceptions were made when (1) the number of observations expected in each cell fell below five, and (2) when some ranges associated with the normal distribution fell into the domain of negative concentrations, which is unrealistic. In the former case, the number of ranges was decreased to five; in the latter, the ranges with negative concentration were grouped together so that the lowest range represented positive concentrations.

It can be demonstrated that in the limit of large sample size, CS has a chi-square distribution with k-r-1 degrees of freedom, where k is the number of concentration ranges and r is the number of parameters in the model (Hald, 1952). In the case of the normal or log-normal distributions, r = 2. From the distribution, one can test the hypothesis that the observed data fit the model, at a given significance level, by comparing CS to the appropriate critical value of the chi-square distribution.

Shapiro-Wilk Test

CANALA CANALA CANALA CANALA CANALA CANALA

The Shapiro-Wilk statistic is a measure of the deviation of sample data from a normal distribution. In a sense, it measures the linearity of the probability plot of sample data on normal probability paper, but has a rigorous basis in the sense of having a statistic with a known distribution which can be used in hypothesis testing of the normality of the underlying population. Previously, some studies of distributions of water quality data have invoked a linear least-squares procedure to test the linearity of probability plots (using Thomas plotting positions), using the r^2 statistic to evaluate goodness-of-fit of data to an empirically fitted line. The linear least-squares procedure, while attractive because of the availability of computer software, has no rigorous statistical basis because of the absence of a known distribution for the statistic, as the plotting positions are arbitrary, and because of the arbitrary selection of the higher-order least squares model (Shapiro and Wilk, 1965).

Unfortunately, the Shapiro-Wilk statistic invokes a set of normalized coefficients based on the covariance matrix of normal order statistics of size n. These have been tabulated in the statistical literature only for values of n less than fifty, and are not available for larger sample sizes.

Tests of various goodness-of-fit statistics suggest that the Shapiro-Wilk statistic provides a generally superior test against non-normality, while the Kolmogorov-Smirnov and Pearson's chi-square statistic can be effective when modified to take account of the practice of estimating model parameters from experimental data (Shapiro, Wilk, and Chen, 1968; Stephens, 1974). Since the coefficients used in the Shapiro-Wilk statistic are unavailable for sample sizes greater than fifty, this test has not been generally applied to EEWTP data except in special cases where the sample size is small. Instead, the Kolmogorov-Smirnov and Pearson's chi-square tests for goodness-of-fit have been applied to selected parameters and data sets to evaluate the applicability of the normal and log-normal distributions to EEWTP results.

TESTS OF PROBABILITY MODELS ON DATA

Examples of goodness-of-fit tests on the normal and log-normal probability models are shown in Table B.1-1, for data from measurements of the EEWTP finished water during Phase IA. The examples shown describe distribution tests on chloride, total chlorine, chloroform by purge-and-trap, gross beta, TOC by composite sampling, total coliforms, TOX, manganese, nitrate + nitrite, and arsenic. In these examples, the chi-square test has been based on conversion of the data into histograms with ten segments of equal probability, except where there are fewer than fifty measurements, in which case five segments are used, or where the detection limit provides a cutoff to the size of a segment.

These results show no systematic pattern to indicate preference of the normal distribution over the log-normal or vice-versa. Both models are rejected at the five percent level for TOC, total chlorine, total coliforms, TOX, manganese, nitrate plus nitrite, and arsenic. A principal reason for this is that the measured data are generally near the detection limit and are therefore reported in the database with only one or two significant digits. The data have been rounded off to eliminate insignificant digits. This act can greatly increase the value of the K-S statistic because the distribution appears "lumpy" with many samples reported at individual concentrations.

The normal distribution tends to be the better model for chloride, chloroform, and nitrate plus nitrite; the log-normal distribution is a better model for gross beta, total coliforms, TOX, manganese, and arsenic. Probability plots of the actual data, showing the 95 percent confidence band of the modified Kolmogorov-Smirnov test, are shown in Figures B.1-1 to B.1-9. The band corresponds to an offset on either side of the modeled distribution equal to the critical value of the modified K-S test at the 0.05 significance level. If any data fall outside the band, the model is rejected at that significance level.

As a consequence of tests of the distributions, it was concluded that the lognormal distribution could be used for the purpose of hypothesis testing. The

TABLE B.1-1

GOODNESS OF FIT TEST RESULTS FOR PHASE IA REWIP FINISHED WATER

Parameter		Fit to Normal Distribution	Distribution
Chloride - 244	3	K-S test: D = 0.050	D = 0.078
		Accept	Reject
	2	Chi-squared test: CS = 20.7	CS = 30.9
		Reject	Reject
TOC	3	K-S test: D = 0.168	D = 0.161
167 t a		Critical Value = 0.052 Reject	Reject
	ê	Chi-squared test: CS = 99	CS = 132
		Reject	Reject
Total Chlorine	æ	K-S test: D = 0.190	D = 0.127
		Reject	Reject
	2	Chi-squared test: CS = 683	CS = 535
		Reject	Reject
Chloroform by Purge-and-Trap	3	K-S test: D = 0.12	D = 0.19
		Accept	Accept
	æ	Chi-squared test: CS = 1.4	CS = 5.3
		Accept	Accept
	Ũ	S-W test: W = 0,926	W = 0.903
		Significance level > 0.1 Accept	SL > 0.1 Accept
Gross Beta	3	K-S test: D = 0.166	D = 0.094
		Reject	Accept
	ê	Chi-squared test: CS = 9.2 Critical level = 14.1	CS = 5.3
		Accept	Accept
	<u> </u>	S-W test: W = 0.919	W = 0.967
		Significance level = 0.01 Refect	SL > 0.1



THE STATE PROPERTY SERVICES SECTION OF SECTION

TABLE B.1-1 (Continued)

Standar.

THE PARTY STATES OF THE PARTY O

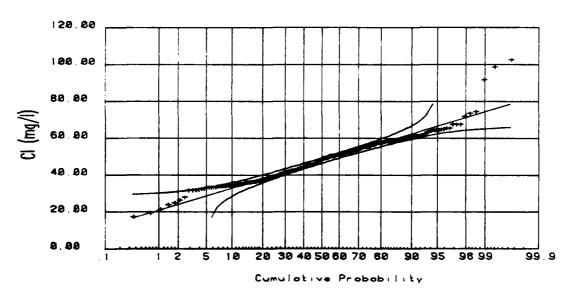
GOODNESS OF FIT TEST RESULTS FOR PHASE IA EEWTP FINISHED WATER

	Parameter		Fit to Normal Distribution1	Fit to Log-Normal Distribution
•	Total Coliforns	3	K-S test: D = 0.26	D = 0.17
			Critical level = 0.00 Reject	Reject
		ē	Chi-squared test: CS = 266 Critical level = 14.1 Reject	CS = 66 Critical level = 11.1 Reject
2	TOX	3	K-S test: D = 0.15	D = 0.12
	662 = u		Critical level = 0.05 Reject	Reject
		ê	Chi-squared test: W = 73	W = 54
			Critical value = 14.1 Reject	Reject
e	Manganese	3	K-S test: D = 0.25	D = 0.11
	63° F		Critical sevel = 0.05 Reject	Reject
		ē	Chi-squared test: CS = 255	CS = 61
			Critical level # 14.1 Reject	Reject
	Nitrate plus Nitrite	3	K-S test: D = 0.11	D = 0.21
	603 1 K		Critical level = 0.05 Reject	Reject
		ê	Chi-squared test: CS = 56	CS = 379
			Critical level = 14.1 Reject	Reject
10.	Arsenic 220	3	K-S test: D = 0.41	D = 0.07
	613-1		Critical level = 0.03 Reject	Reject
		ē	Chi-squared test: CS = 870 Critical level = 14.1 Reject	CS = 37 Critical level = 9.5 Reject

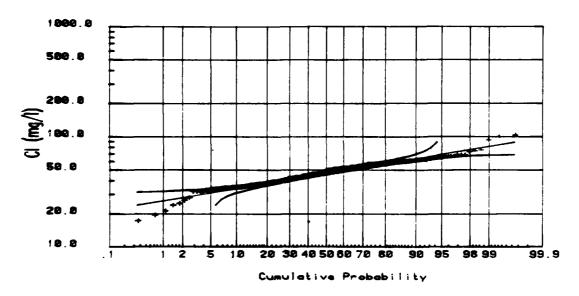
^{1.} All tests and critical values evaluated at the 0.05 significance level.



A SAME



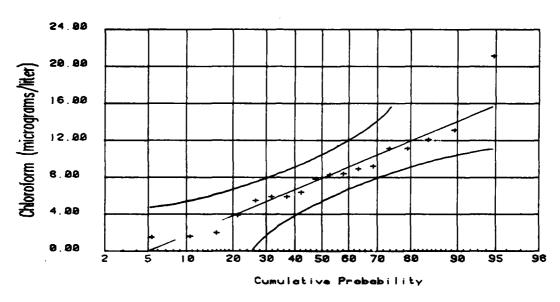
(a) Normal probability distribution



(b) Log-normal probability distribution



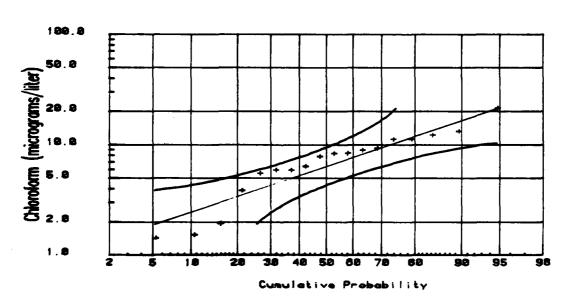
CHLORIDE IN EEWTP FINISHED WATER
95 PERCENT CONFIDENCE BANDS OF MODIFIED K-S TEST
FIGURE B. 1-1



and which are not been been

THE LABORAL MAKERS RESIDENT WINDOWS SECURE

(a) Normal probability distribution

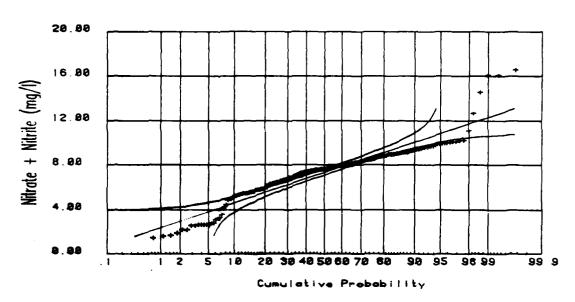


(b) Log-normal probability distribution

CHLOROFORM (VOA) IN EEWTP FINISHED WATER
95 PERCENT CONFIDENCE BANDS OF MODIFIED K-S TEST
FIGURE B. 1-2

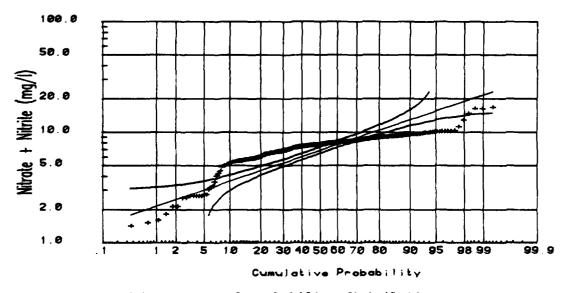






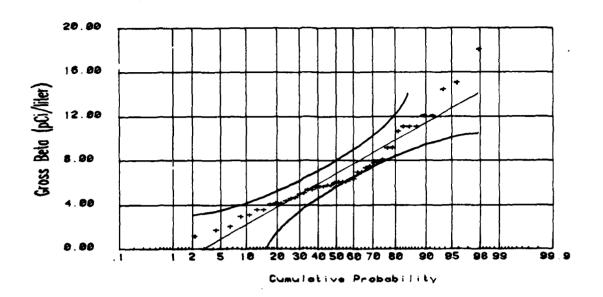
(a) Normal probability distribution

ALL MOROR COUNTY DIVINE LANGE CANADA SANDA CONTRA MOROR ANDROS MOROS



(b) Log-normal probability distribution



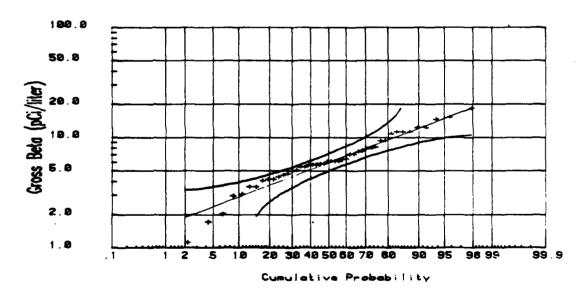


(a) Normal probability distribution.

SACT SECULIAR STATE SECULIAR S

AN AND

THE PROPERTY OF THE PROPERTY O



(b) Log-normal probability distribution.

GROSS BETA IN EEWTP FINISHED WATER

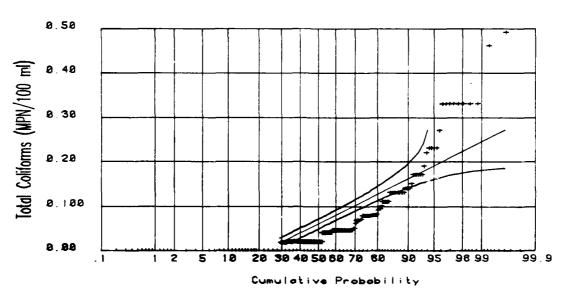
95 PERCENT CONFIDENCE BANDS OF MODIFIED K-S TEST
FIGURE B. 1-4



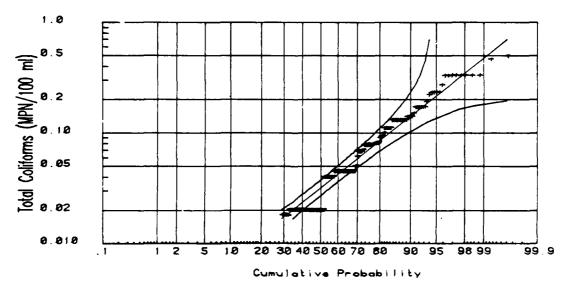








(a) Normal probability distribution



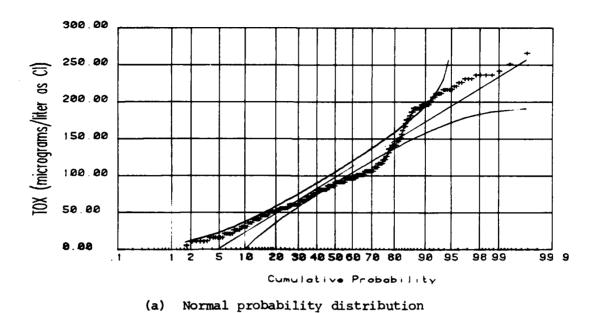
(b) Log-normal probability distribution

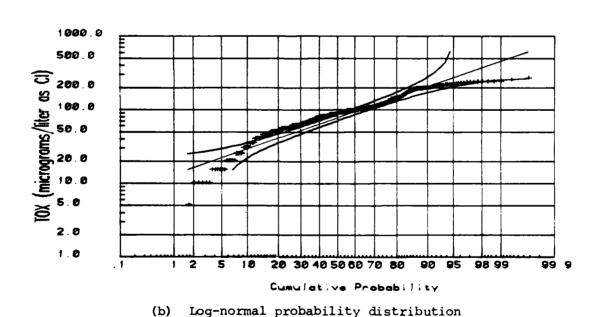


ASSESSED AGENCIAL INCOME. INCOME.

TOTAL COLIFORMS IN EEWTP FINISHED WATER

95 PERCENT CONFIDENCE BANDS OF MODIFIED K-S TEST
FIGURE B. 1-5

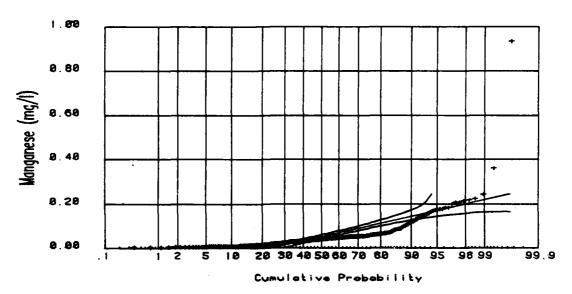




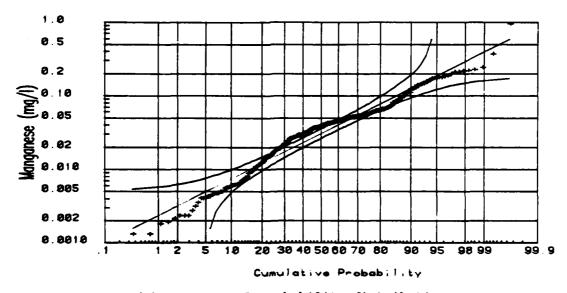
TOTAL ORGANIC HALIDES IN EEWTP FINISHED WATER
95 PERCENT CONFIDENCE BANDS OF MODIFIED K-S TEST
FIGURE B. 1-6







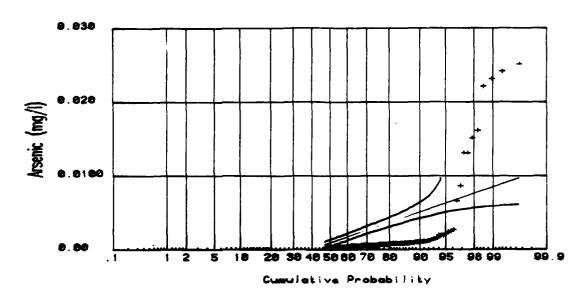
(a) Normal probability distribution



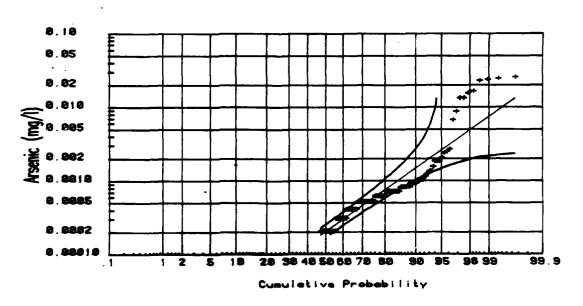
LANGERS SURSESSON MARKETS INSURANCE SESSIONS

(b) Log-normal probability distribution





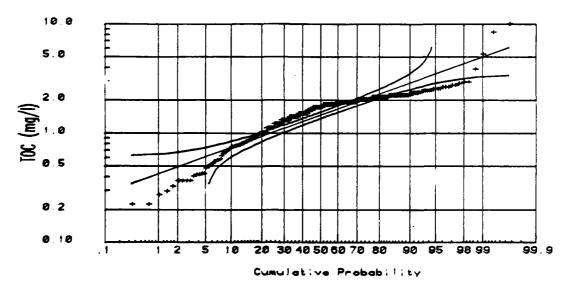
(a) Normal probability distribution



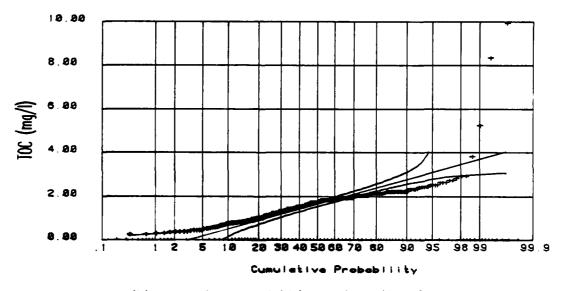
(b) Log-normal probability distribution

ARSENIC IN EEWTP FINISHED WATER
95 PERCENT CONFIDENCE BANDS OF MODIFIED K-S TEST
FIGURE B. 1-8





(a) Log-normal probability distribution



(b) Normal probability distribution





basis for this was threefold. (1) The error associated with the use of geometric mean and spread factor for t-tests, where the normal model fits the data better than the log-normal model, is smaller than the error associated with the reverse case (use of arithmetic mean and standard deviation in t-testing on log-normally distributed data), because of the significance of concentrations skewed to higher values in the highly skewed log-normal distribution. (2) The basis of calculation used for arithmetic means included an arbitrary assumption when concentrations were below the detection limit which renders these estimates less useful for hypothesis testing than estimates of geometric means based on the maximum likelihood principle. (3) In cases where neither normal nor log-normal distribution can be shown to pass a goodness-of-fit test, the use of the log-normal distribution yield results which are less sensitive to "outliers;" the geometric mean is less affected by occasional high values than the arithmetic mean, and therefore has more robust characteristics when considering the possibility of sample contamination or lab error.

STATISTICAL TECHNIQUES FOR EVALUATING DATA AND ESTIMATING SAMPLING REQUIREMENTS

When the normal distribution is used as the statistical model for a water quality parameter, the arithmetic mean and standard deviation (or variance) of the distribution are estimated from sample data. Similarly, when the log-normal distribution is used as a model, the underlying population is characterized by its geometric mean and spread factor, using sample data. The techniques for estimating model parameters from samples are universally known and used in the field of statistics in the usual case when data are measured during random sampling. In the special case where some data can not be reported because they fall below a known detection limit, other approaches are available. These approaches, which are based on the principle of maximum-likelihood estimation, are described in this section. Another complication is the case when sampling is not truly random, but is performed in a systematic manner over time, which results in measurements which are not necessarily independent of each other. This case, of auto-correlation in time-series sampling, is also discussed.

In the discussion which follows, reference is made to the concept of "uniformly minimum variance unbiased" estimates of model parameters. This refers to two desired properties of the "best" estimator. First, the ideal estimate of a parameter should have a minimum difference from the true value of the parameter. This difference can be measured by the mean squared error, which is the expected value of the square of the difference between true population parameter and its estimate. A uniformly minimum variance estimate is one which has a lower mean squared error than all other estimates.

The second desirable property of an estimate is that the expected value of the estimate be equal to the true population parameter. In such a case, the "bias" of the estimate is zero and the estimate is said to be unbiased. It does not consistently overestimate or underestimate the value of the parameter. The concept of a uniformly minimum variance unbiased (U.M.V.U.) estimate is central to the basis of parameter estimation.

THE PARTY OF THE PROPERTY OF THE PARTY OF TH

Another concept which figures significantly in the discussion of results is the "maximum likelihood estimate." This is an estimate of a parameter which is "most likely" to have produced the data which have been sampled. An infinite number of values for a parameter can have given rise to a particular set of observations; each value of the parameter has some likelihood of doing this. The maximum likelihood estimate (M.L.E.) is that number, calculated from the sample data, which has the highest likelihood of any of the possible values of the parameter which could have led to the sample results.

PARAMETER ESTIMATION: DATA ALL QUANTIFIED

When concentration values in a data set are all reported, i.e., when there are no "Not Detected" values, the estimation of the parameters of the underlying population is straightforward. For the hypothesis that the population is normally distributed, the uniformly minimum variance unbiased (U.M.V.U.) estimator of the population mean, µ, is simply the sample mean,

$$\bar{x} = \frac{1}{n} \sum_{i=1}^{n} x_i \tag{6}$$

Similarly, the U.M.V.U. estimator of the population variance is the expression

$$s^{2} = \frac{1}{n-1} \sum_{i=1}^{n} (x_{i} - \bar{x})^{2}$$
 (7)

Note that the denominator of this expression is n-1 rather than n; this is the unbiased estimator of variance. The maximum likelihood estimate of variance has a similar form but uses a denominator of n, and is a biased estimate.

For estimation with a hypothesized log-normal model, the U.M.V.U. estimator of the population geometric mean is the sample geometric mean,

$$M = \begin{bmatrix} n \\ \overline{II} \\ i=1 \end{bmatrix} y_i$$

$$= 10$$

$$1/n \sum_{i=1}^{m} \text{Log } y_i$$

$$= 10$$
(8)

The estimator of the population spread factor is the sample spread factor,

$$s = 10^{\left(\frac{1}{n-1} \sum_{i=1}^{n} (\log_{10} y_i - \log_{10} M)^2\right)^{\frac{1}{2}}}$$
 (9)

These estimates are strictly analogous to those of the normal distribution, transformed logarithmically.

CHARACTERIZATION OF PROCESS EFFLUENTS AND FINISHED WATER

The EEWTP process waters are characterized statistically in terms of probability model parameters; for the finished water, this information is used in evaluating water quality against specific objectives. Definition of the objectives was based on a review of the water quality impacts of given parameters such as long and short term health effects criteria. For example, to evaluate potability with respect to a given parameter, the measure of central tendency in the EEWTP effluent is compared against water quality goals or against frequency distributions for alternate drinking water sources such as the three local water treatment plants. Alternatively, the behavior of a given parameter at the high end of the probability distribution can be characterized to assess potential health risks associated with toxic effects of infrequent elevated concentrations. The purpose of this exercise is to define descriptive statistical parameters which could be used in evaluating these objectives. Needless to say, the development of quantitative descriptions of the potability of drinking water supplies is currently open to much debate.

Procedures were developed for characterization of the geometric mean (complete and incomplete sample sets), hypothesis testing, comparison of finished waters and characterization of frequency criteria. These are described below. Also, at the beginning of the second year of sampling, procedures were developed to estimate the sampling frequency for each parameter believed necessary to adequately characterize its value.

In many cases, the calculations are based on inversion of a t-test used in calculating confidence intervals or testing a hypothesis at a given significance level. The t-distribution in these cases is robust to non-normality, in that the procedures are usually applicable to data which are not necessarily distributed normally (or log-normally in the t-test on logarithmically transformed data), so that the results of these calculations do not depend on the exact shape of the underlying population distributions, as described by Box, Hunter, and Hunter (1978).

Characterization of the Geometric Mean - Data Sets with Values All Positive

When there are no values in a data set which fall below the detection limit, it is straightforward to calculate the population geometric mean and to establish confidence intervals around the estimate. A log-normal population distribution is assumed.

Let
$$x = \log_{10} y$$
 (10)

where

MACHER PRINTERS SERVING KARREES DONNY (SS

STREET COORDS | CONTROL | CONTROL | CONTROL

y = concentration n = number of samples

$$\bar{x} = \frac{1}{n} \sum_{i=1}^{n} x_{i} \tag{11}$$

$$s^{2} = \frac{1}{n-1} \sum_{i=1}^{n} (x_{i}^{-x_{i}})^{2}$$
 (12)

where

x = estimate of the population average of the logarithm of concentration

s = estimate of the population standard deviation

Confidence Bounds. The estimate \bar{x} is distributed t_{n-1} , so we can establish a 95% confidence interval about \bar{x}

$$\left(\bar{x} - \frac{t_{n-1} (0.975)s}{(n)^{\frac{1}{2}}}\right) \leq \mu \leq \left(\bar{x} + \frac{t_{n-1} (0.975)s}{(n)^{\frac{1}{2}}}\right)$$
(13)

or simply

$$\frac{1}{x} \pm \frac{t_{n-1} (0.975)s}{(n)^{\frac{1}{2}}}$$
 (14)

where

 $t_{n-1}(0.975) = 97.5$ percentile value of the t-distribution with n-1 degrees of freedom and $\bar{x} = \log_{10}$ (geometric mean) or

$$M = 10^{\overline{X}} \tag{15}$$

$$S = 10^{S} \tag{16}$$

where

M = population geometric mean S = population spread factor

Therefore, the 95 percent two-sided confidence interval about the true population geometric mean is

$$\frac{-t_{n-1} (0.975)}{(n)^{\frac{1}{2}}} \qquad \frac{t_{n-1} (0.975)}{(n)^{\frac{1}{2}}}$$
(M) (s) $\leq \mu_{g} \leq (M)$ (s) (17)

where $t_{n-1}(0.975)$ is the 97.5 percentile of the t-distribution with n-1 degrees of freedom.

Sample Size. One can choose a sample size n* required to restrict this confidence interval within arbitrary limits. For example, one could aim to estimate M within a factor of two, i.e., between 0.707M and 1.414M. That is, the value of

$$\frac{t_{n-1}}{s} \frac{(0.975)}{(n)^{\frac{1}{2}}}$$
(18)

is set equal to (2) 1/2 = 1.414 so that the ratio of the upper bound to the lower bound is two. If the geometric mean for a given water quality variable is significantly below the goal, and no single sample exceeds the goal, then the minimum sample size n^* could be chosen to attain this arbitrary accuracy about M. Since preliminary estimates were obtained for S for each parameter at the beginning of the second year of sampling when decisions were made about sample frequency, an estimate of n^* was made by setting

$$\frac{t_{n^*-1}}{s} = (2)^{\frac{1}{2}}$$
 (19)

and solving for n* for each parameter. This procedure is applicable to parameters where all values are above the detection limit. Note that this sample size has not been corrected for serial correlation, discussed below.

Characterization of the Geometric Mean - Values Below Detection Limit

For parameters with data sets which have concentrations below the limit of detection, it is not possible to evaluate the sample geometric mean as described above. One way to deal with this is to estimate the sample geometric mean and spread factor by fitting a linear regression to the log-normal probability plot. An example is shown in Figure B.1-5(b) where data for total coliforms are plotted on log-normal probability paper using Thomas plotting positions. The plotting positions are based on setting the probability coordinate of each point to i/(n+1), where i is the rank of the point in ascending order, and n is the total number of points. This convention is useful because it does not assign plotting positions of 0 or 100% to points, and is symmetrical. However, it is also arbitrary.

A linear least-squares regression of the data against plotting positions gives a "best-fit" line. This line has an intercept at the fifty percentile axis of 0.035/100 ml, corresponding to an estimate of geometric mean, and a slope which corresponds to a spread factor of 3.94. The main drawback of this approach is that the estimates have no rigorous statistical basis, and the use of Thomas plotting positions is arbitrary. This approach was therefore used only to estimate a required sample size.

As in the previous procedure, one can calculate a sample size to estimate M_e with 95 percent confidence intervals within a range of two. For example:

$$\frac{t_{63} (0.975)}{(64)^{\frac{1}{2}}} = 1.414$$
 (20)

so that $n^* = 64$ samples.

THE PERSON IN THE WAY WINDOWS THE WAY WINDOWS WINDOWS

A second alternative approach for dealing with values below the limit of detection is to calculate the geometric mean and spread factor (M_p, S_p) for the subset of samples which have quantifiable values. This approach excludes information from the estimate of geometric mean and spread factor, and also tends to greatly overestimate the geometric mean. This approach was also used only to calculate required sample size.

A required subset size (of positive values) was estimated by then calculating the number of samples with quantifiable values required to estimate M_p with 95 percent confidence intervals within a range of two. From this subset size a total required sample size was calculated by multiplying by the ratio of total samples to positive samples in the existing data base. This frequency criterion gave an estimated total sample size required to characterize the quantifiable values.

An example of this calculation at the beginning of the second year of sampling was total coliforms, where the geometric mean and spread factor of the quantifiable values were $M_p = 0.0636/100 \text{ ml}$ and $S_p = 2.702$. Out of a total of 223 samples taken, 165 samples had quantifiable values. Using the previously described procedure, the subsample of quantifiable values, N_p , required to characterize M_p and S_p were calculated by noting that

$$\frac{t_{34}^{(0.975)}}{(35)^{\frac{1}{2}}} = 1.414$$
(21)

where $t_{34}(0.975)$ is the 97.5 percentile value of the t-distribution with 34 degrees of freedom, so that $n_p = 35 = \text{number of quantifiable samples required}$ to characterize M_p . Then an estimate of the total number of samples required was

$$\frac{(35)(223)}{165} = 46 \tag{22}$$

A third approach to dealing with data sets with values below the detection limit is to estimate geometric mean and spread factor based on the principle of maximum likelihood estimation. This is a commonly used statistical approach where the estimates of the parameters of a probability model are set equal to the values which have the highest likelihood of giving rise to the observed data. In the case of "censored data" where the concentrations below the detection limit are known to lie in a range between zero and the detection limit, but for which the actual concentrations are unknown, it is possible to develop a rigorous basis to make this estimate.

The case of data below detection limit is a special case of the general problem of parameter estimation with censored, or incomplete, data. The principles of the censored data problem with the normal probability model are described by Hald (1949), Grundy (1952), Cohen (1957), Blight (1970) and Dempster et al. (1977). For the normal probability model, the likelihood function which



Section Straigs I were presented

describes the probability of obtaining a given outcome by drawing a censored sample from a population with mean μ and variance 2 is a straightforward analytic function. A maximum of this likelihood function can be achieved by setting the derivatives of the likelihood function with respect to μ and to equal to zero. These normal equations may be solved by numerical or graphical methods or by tabulated solutions. In the case of the analysis of EEWTP data, an iterative method was used to solve the normal equations in order to estimate the values of population geometric mean and spread factor with the highest likelihood of producing observed concentrations.

Consider the case of a data set with N observations, of which n observations $(x_1, x_2,..., x_n)$ are greater than or equal to the detection limit D, and m observations are below the detection limit. If the population mean and standard deviation are μ and σ , respectively, then the probability of obtaining an individual positive observation with concentration x is

$$f(x) dx = \frac{1}{\sqrt{2\pi}\sigma} exp\left(-\frac{1}{2\sigma^2}(x-\mu)^2\right) dx$$
 (23)

The probability of obtaining a value below the detection limit is

$$P(x$$

and the likelihood function of obtaining the observed sample is

$$L = \begin{pmatrix} n \\ \overline{||} & f(x_i) \\ i=1 \end{pmatrix} \left(p(x < D) \right)^{m}$$
 (25)

By setting the derivatives $\partial L/\partial \mu$ and $\partial L/\partial \mu^2$ equal to zero, the likelihood function may be maximized. As a result of this, the two normal equations can be obtained:

$$\mu = \frac{1}{N} \sum_{i=1}^{n} x_i + \frac{m}{N} \frac{\int\limits_{-\infty}^{D} x \exp\left(-\frac{1}{2\sigma^2} (x-\mu)^2\right) dx}{\int\limits_{-\infty}^{D} \exp\left(-\frac{1}{2\sigma^2} (x-\mu)^2\right) dx}$$
(26)

$$\mu^{2} + \sigma^{2} = \frac{1}{N} \sum_{i=1}^{n} x_{i}^{2} + \frac{m}{N} \int_{-\infty}^{\infty} x^{2} \exp\left(\frac{1}{2\sigma^{2}} (x-\mu)^{2}\right) dx$$
 (27)

The approach taken for EEWTP data has been to use a recursive set of equations to iteratively estimate the values of mean and standard deviation, having made initial estimates μ_1 and σ_1 :

$$\mu_{i+1} = \frac{1}{N} \sum_{i=1}^{n} x_{j} + \frac{m}{N} \mu_{i} - \frac{\sigma_{i}m}{N} \int \frac{f\left(\frac{D-\mu_{i}}{\sigma_{i}}\right)}{\int \frac{D-\mu_{i}}{\sigma_{i}}}$$
(28)

$$\sigma_{i+1}^2 = \frac{1}{N} \sum_{i=1}^{N} x_j^2 - \frac{n}{N} \mu_{i+1}^2 + \frac{m}{N} \sigma_i^2 - \frac{\sigma_i m}{N}$$
 (29)

This maximum likelihood procedure has been used only for estimating geometric mean and spread factor by performing the recursive solution on logarithmically transformed concentration data. The results have been quite successful in terms of convergence of the recursive algorithm and in the approach of the resulting estimates to known mean and standard deviation in censored synthetic data. Similarly, the apparent fit to observed distributions of real data seems adequate on the basis of probability plots, as in Figures B.1-1 to B.1-9.

Hypothesis Testing - Comparison With a Goal

For a normal distribution of logarithmically transformed data, the appropriate statistic for testing the hypothesis that the population geometric mean falls below a goal or standard is a t-test of n-1 degrees of freedom. Using the logarithmic transformation as before, the null hypothesis is that

$$\mu < x$$
 goal (30)

or,
$$\left(\mu_{g} = 10^{\mu}\right) < \left(10^{\frac{x}{\text{goal}}} = y_{\text{goal}}\right)$$
 (31)

and the appropriate statistic used to disprove this is

$$T = \frac{n^{\frac{1}{2}} (x_{goal} - \overline{x})}{s}$$
 (32)

where

MANAGE THE STATE ARREST AND A PARTY PARTY

$$s = \left(\frac{1}{n-1} \sum_{i=1}^{y} (x_i - \overline{x})^2\right)^{\frac{1}{2}}$$
 (33)

The null hypothesis is rejected at the five percent significance level if

$$T > t_{n-1}$$
 (0.95) (34)

An example of a hypothesis test against a goal is the comparison between the level of arsenic in EEWTP finished water during Phase IA and the EPA maximum contaminant level of 0.05 mg/L. The geometric mean (as estimated by the maximum likelihood procedure using the detection limit of 0.0002 mg/L) is 0.000207 mg/L and the spread factor is 4.65. 279 samples were collected and analyzed. The logarithms of the arithmetic mean and standard deviation of arsenic concentration are -3.684 and 0.667. So the t statistic for the comparison to this goal is:

$$T = \frac{(279)^{\frac{1}{2}}(\log_{10}(0.05) - \log_{10}(0.00207))}{\log(4.65)} = 34.6$$
 (35)

This value is much larger than the critical value of the ^t278 statistic at the five percent significance level, 1.98. Therefore the null hypothesis that the geometric mean arsenic concentration is equal to the goal is rejected. This test demonstrates that the geometric mean is significantly below the EPA standard at the five percent significance level.

Given estimates of x and s one can calculate a value of n adequate to show that the population mean is significantly below the goal:

$$(n^*)^{\frac{1}{2}} \ge \frac{\operatorname{st}_{n^*-1}(0.95)}{\operatorname{x}_{\operatorname{goal}} - \overline{\mathbf{x}}}$$
 (36)

An example of this calculation is the arsenic case, for which n* is set such that

$$(n^*)^{\frac{1}{2}} \ge \frac{\log_{10} (4.65) t_{n^*-1} (0.95)}{\log_{10} (0.05) - \log_{10} (0.00207)} = 0.48 t_{n^*-1} (0.95) (37)$$

The smallest value of n* satisfying this condition is 3. This indicates that, if the spread factor is adequately characterized, only a few samples are needed to demonstrate that the geometric mean for arsenic is significantly below the goal. However, it is clear that without the previous data, it would be impossible to characterize the spread factor to a degree sufficient to prove the hypothesis. with only four samples.

Hypothesis Testing - Comparison of Finished Waters

THE PARTY OF THE PARTY OF THE PARTY AND THE PARTY OF THE

For some parameters it is possible to show that the EEWTP finished water has concentrations significantly below values in the treated waters of the local water treatment plants. The procedure for this is the t-test for the difference

	(D-01)	36 865	OPE	POTION	μοίν	TENONC	E OND	DEDEN	MONCE	EUQ! II	OTION	OF THE			
ľ		SIFIE	POT ENG	INEERS	S INC I	PASADE	U) MON NA CA 31-80-	JMI	10NTGO	EVALU MES M) METRY	CONSU SEP 83 F/G 1	3	NL	6 .	`\
	•														
L															ل



PARCE CONTRACTO LANGERS. CATEGORIES CONTRACTOR

MICROCOPY RESOLUTION TEST CHART

NATIONAL BUREAU OF STANDARDS-1963-A

ny kaon**a.** Taona amin'ny in means of two normal populations, valid when the variances of the two populations are assumed equal. Consider measurements $X_1, ..., X_n$ from one site, and $Y_1, ..., Y_m$ from another site, with sample means \overline{X} and \overline{Y} . If the variances of the two populations are assumed equal, then the appropriate test of equivalence between populations is a t-test with null hypothesis that the population means are equal. The test statistic:

$$T = \left(\frac{nm}{n+m}\right)^{\frac{1}{2}} \left(\frac{y-x}{y-x}\right)$$
 (38)

where

CAMPAGE CAMPAGE LACESCASS CONTRACTOR

$$s_{2} = \frac{1}{n+m-2} \left[\sum_{i=1}^{n} (x_{i} - \overline{x})^{2} + \sum_{j=1}^{m} (Y_{j} - \overline{Y})^{2} \right]$$
(39)

is t-distributed with n-2 degrees of freedom. The two-sided test at the 0.05 significance level rejects the null hypothesis if

$$|T| \ge t_{n+m-2}(0.975)$$
 (40)

If it is not possible to assume that X and Y have equal variance, then Welch (1938) has shown that an appropriate t-test on the hypothesis of equal means can be performed using

$$T = \frac{\overline{Y} - \overline{X}}{-\sqrt{\frac{1}{n(n-1)}} \sum_{i=1}^{n} (x_i - \overline{X})^2 + \frac{1}{m(m-1)} \sum_{j=1}^{m} (y_j - \overline{Y})^2}$$
(41)

where T is approximately t-distributed with k degrees of freedom, where

$$k = \left[\frac{c^2}{n-1} + \frac{1-c^2}{m-1}\right]^{-1}$$
 (42)

$$c = \frac{\frac{1}{n(n-1)} \sum_{i=1}^{n} (x_i - \overline{x})^2}{\frac{1}{n(n-1)} \sum_{i=1}^{n} (x_i - \overline{x})^2 + \frac{1}{m(m-1)} \sum_{j=1}^{m} (y_j - \overline{y})^2}$$
(43)

These tests are useful for parameters in which the mean value in the EEWTP finished water is not far below the goal, but the mean values for the alternate water sources are higher. They are also useful where no goal for a parameter has been set, but health risk is an issue. One can also calculate a sample size for the EEWTP finished water and the other sources required to prove a significant difference, by an inversion of the t-test similar to that taken for the EEWTP comparison to a goal.

Characterization of Frequency Criteria

It is often relevant to characterize the frequency with which a goal or standard is exceeded. This is especially applicable to parameters for which acute toxicity or aesthetic quality are associated with the goal. Using the method of frequency substitution by which unknown population frequencies are replaced by observable sample frequencies, the maximum likelihood estimate for the probability $P(Y>Y_{goal})$ is given by the fraction of samples observed to exceed the goal in the existing database. This approach to the problem discards the difficulties and added information associated with finding an adequate lognormal distribution and instead uses a binomial trial model in which the concentration either exceeds the goal or it doesn't, with fixed probabilities for either outcome. However, because it discards useful information it tends to give estimates with lower confidence than those based on the log-normal model.

Random variable X is observed in n samples. The probability that the concentration X_i exceeds the goal (X_{gQal}) is equal to θ . The probability that $X_i \leq X_{goal}$ is $1-\theta$. The estimate for θ is Z, where

$$\overline{Z} = \frac{1}{n} \sum_{i=1}^{n} Z_{i}$$
 (44)

where

CONTRACTOR CONTRACTOR

$$\begin{array}{ll} Z_i = 0 & \text{if} & Y_i \leq Y_{goal} \\ Z_i = 1 & \text{if} & Y_i \geq Y_{goal} \end{array} \quad \text{and} \quad$$

The variance of Z is equal to $\theta(1-\theta)$. The estimate for the variance is then $\vec{Z}(1-\vec{Z})$. It is possible to calculate exact confidence limits about θ using tables. For example, since 3 out of 279 samples for mercury in Phase IA finished water exceeded a water quality goal of 0.002 mg/L, the estimate for the probability that the goal is exceeded is 3/279 = 1.08%, with a ninety-five percent confidence interval calculated to be 0.223 to 3.08 percent. Another way to look at this is to observe that the 97.5 percent one-sided upper confidence limit is the region $\theta < 3.08\%$.

By assuming that \bar{Z} measured in preliminary data will be valid for future samples, the number of samples required to narrow the upper confidence limit to an arbitrary bound can be calculated. In this example, one might in theory calculate an estimate for the sample size N required to place a 95 percent upper confidence limit of, say $\theta < 5$ percent or $\theta < 10$ percent.

When no samples are observed to exceed the goal, the method of frequency substitution gives estimates of θ equal to zero. In this case the 95 percent upper confidence limit is given by $\theta = 1 - (.05)^{1/n}$. We can set the upper confidence limit to an arbitrary level, say ten percent, and calculate a sample size: n = 29 for 10 percent, n = 59 for 5 percent. For cadmium in finished water during the EEWTP Phase IA, no samples out of 279 exceeded the federal MCL, so the 95 percent upper confidence limit is a 1.07 percent frequency of

exceeding the MCL. This is one way to set a minimum sample size for those parameters which seem to fall clearly below the goal.

Serial Correlation Correction

The use of hypothesis tests and confidence bounds, as described above, apply to sample data which represent independent, random observations of a random variable. When sampling takes place on a routine schedule, the samples are not necessarily random and independent because the level measured on one day may be partially dependent on previous days' levels. If $\rho(k)$ is the lag-k autocorrelation coefficient for the population, defined by

$$\rho(k) = \frac{\sum_{i=1}^{n} (x_i - \bar{x}) (x_{i+k} - \bar{x})}{\sum_{i=1}^{n} (x_i - \bar{x})^2}$$
(45)

then it can be shown that the variance of the sample mean is equal to

$$\frac{\sigma^2}{n} \left[1 + 2 \sum_{k=1}^{n-1} \frac{n-k}{n} \rho(k) \right]$$
(46)

whereas the variance of the sample mean for independent samples is σ^2/n . That is, the variance in this estimate of the mean is larger when the samples are not random and independent; the confidence in this estimate of the mean is poorer than if the samples were independent. A correction factor, C, equal to the term in brackets in equation 46, may be multiplied by sample size n, in order to achieve variance in the mean equivalent to n independent samples. One may define effective sample size:

$$n_{\text{eff}} = \frac{\frac{n}{n-1}}{\frac{n-1}{n}} = \frac{n}{C}$$

$$1 + 2 \sum_{k=1}^{\infty} \frac{n-k}{n} \rho(k)$$
(47)

In other words, the sample sizes calculated by previous procedures can be multiplied by C to obtain a corrected sample size. An estimate for C can be calculated using sample serial correlation coefficients and the existing data set. For example, the serial correlation coefficients for the existing time series of mercury at the time when the year two sample program was being developed were:

lag 1 day = .186 lag 2 days = .217 lag 3 days = .100 lag 4 days = .117 lag 5 days = .079

and higher lags had insignificant correlation. These values yielded a correction factor C = 2.36; therefore the effective sample size is approximately n/2.36.



THE INTEREST TOWNERS INTORNED INTORNESS TOWNERS TOWNERS TOWNERS TOWNERS TOWNERS THE

SECTION 2

PROJECT DATA MANAGEMENT

Over 500,000 data records were generated over the course of the three year EEWTP project. This data originated from plant operations monitoring, water quality analysis, and engineering testing.

The purpose of the project data management system was to provide an efficient, rapid capability for storage, retrieval, and analysis of these data. The data management system involved the use of on-site and off-site computer hardware with software specifically developed for the input and presentation of such vast quantities of data.

HARDWARE

OFF-SITE

The project utilized two off-site computers. The main computer is a Digital Equipment Corporation (DEC) VAX 11/780 operating under the Virtual Memory System (VMS) operating system. In its configuration at the time of project termination, it had 4 Mega bytes (Mb) central memory, a 67 Mb RM03 disc drive and three 256 Mb RM05 disc drives. Peripherals included a tape drive, card reader, line printer, drum plotter, and 56 communications ports supporting directly connected and remote terminals. A Hewlett-Packard 3354 B Laboratory Automation System was used to integrate instrument data and manage the sample inventory. The Hewlett-Packard 3354 B Laboratory Automation System was used for data collection, storage, analysis and report generation, as well as for management of laboratory time and resources. It is a disk-based system that is capable of controlling and gathering data from up to sixteen laboratory instruments simultaneously while operating a multi-terminal BASIC interpreter that provides general purpose computing and high resolution graphics capabilities.

The system is connected via HP18652A analog-to-digital modules to all laboratory instruments that produce an analog signal and is used to collect, store, and quantitate the data and to generate reports. Hardcopy reports are produced on the HP2631G graphics printer and on an HP2635A printing terminal. Raw data is stored on disk, and a link to the VAX 11/780 provides access to magnetic tape.

ON-SITE

SCHOOL COMPANY SERVICE

One computer was used for on-site data entry, a Digital Equipment Corporation MINC computer. Both the MINC and the VAX computers are built around Digital's PDP-11 series of computers. The MINC is configured with an RX02

floppy disk drive, providing 1 Mb of storage. The addition of a DLV11-E asynchronous line interface provided a means of connecting directly with the VAX 11/780.

Three video display terminals were utilized on-site, including one with enhanced graphics capability. Additional hardware devices included one printer terminal used primarly as a printer, a digital plotter, and a video hardcopy unit. The terminal devices were multiplexed with a statistical multiplexer which was connected to a leased digital telephone line.

SOFTWARE

DATABASE CONCEPT AND DESIGN

Management of the data from this project was a complex task due to the large number and diverse sources of data collected. The development of computer software was divided into three distinct phases: systems analysis, system design, and system construction.

Systems Analysis

The systems analysis phase consisted of an overall evauation of the data management program, with a detailed analysis of user requirements. Several demands were placed on the data management system which were difficult to satisfy with traditional civil engineering (or even traditional computer science) database technology. Some of the basic aspects of the data which determined the final system design are listed below:

- 1. The data would cover a wide range of physical, chemical, microbiological, engineering and operational parameters.
- 2. Sampling would be done at numerous locations on-site as well as at three local water treatment plants.
- 3. Data had to be input and accessed from the EEWTP on-site and Pasadena off-site laboratories, which are separated by more than 3,000 miles.
- 4. Three different computers would be used, two of them manufactured by Digital Equipment Corporation and one by Hewlett-Packard.
- 5. The system had to be flexible, that is, it had to accommodate changes in the nature and frequency of sample collection, some of which could not be foreseen at the start of the project.
- 6. The background and specific needs of potential users of the system were quite diverse. Engineers and scientists as well as management personnel would all potentially desire access to the project data for their own specific interests and applications.

These demands were studied in detail during the systems analysis phase and a general concept for management of the project data was developed.

System Design

The Pasadena VAX 11/780 was used for storage and access to the project database. Some data was collected by the other computers, but the VAX was ultimately the central processor. This allowed central access to the database for laboratory and engineering personnel from both laboratory locations, using direct access lines.

The fundamental concept for all database architecture and structure was an indexed sequential file system which provided quick and convenient data storage and access. A specification for this structure was prepared at the beginning of the design phase and served as the overriding criteria for all project software development.

With the indexed sequential access method of file organization, each data point or result corresponds to a record or line of data. Therefore for each data point or result, there is a corresponding record in the file (Figure B.2-1). Each record consists of three major components, the sample identification code, the parameter code and the value or result. The sample identification code serves as a tag or label which identifies the sampling location and sampling time of that piece of data. Similarly, the parameter code is a number that identifies which analysis was conducted. By using software available on the VAX, the user had the capability to sort through the database very quickly and retrieve only those records of interest by narrowing the search to selected parameters, sampling locations or times.

The primary benefit to this approach for organizing the database is related to the efficiency of searches for individual records or groups of related records. The designer of the database has an option of selecting one or more fields (for example, parameter code and/or location) within each record as a key or index. A table of contents of these indices is maintained for all stored project records.

Whenever the VAX is instructed to retrieve records from the indexed database, it goes first to the table of contents, looks for the correct index, and then goes directly to the correct location in the file. This type of activity is analogous to searching through the index or table of contents in a large book and going directly to the listed page for desired information rather than having to read the book each time. Needless to say, this approach is much more efficient than an exhaustive search through the entire database and significantly reduces the time and cost of data retrieval.

To support this approach, a standard sample identification code (SID) was developed. It consists of six basic components.



FIGURE B.2-1 DATA MANAGEMENT SYSTEM INDEXED SEQUENTIAL ACCESS METHOD CONCEPT

Sample Identification Code				tion	Parameter Code	Value
P	LLLS	тт	YMMDD	AAAAA	ССССМ	Real Number
R	0762	07	10316	00000	0570	2.3
R	0762	07	10317	00000	0570	2.2
R	0762	07	10316	00000	2040A	1.5
R	0762	07	10317	00000	2040B	17
L					L	
		Cor	† itinued		† Continued	† Continued



MANAGES STANDED CLOSES AND STANDED STA

Program (P)

A one character field which represents the particular program under which a sample or piece of data was collected.

Typical codes:

- R = Routine Water Quality Testing Program
- D = Operator Data Collection System
- T = Testing Program for Process Adjustment and Modification
- S = Startup (Data taken prior to 16 March 1981)
- Q = Quality Control/Quality Assurance

The program codes are used primarily to conveniently group the data, and identify the general sampling program which produced the data.

Location (LLL)

A three character field which identifies the exact location where a sample was collected. Every sample tap at the EEWTP and local WTP where data or samples were collected was assigned a location code. A listing of location codes is provided in Table B.2-1 at the end of this section.

Sampler Type(S)

A single character field identifies the type of sample taken. Examples of sampler types include grab, automatic composite sample, meter or gauge readings. Table B.2-2 at the end of this section contains a list of the sampler types used on the project.

Time (TT)

This is a two character field which identifies the time of sample collection on a 24 hour clock. For composite samples, this is the time the sample was retrieved.

Date (YMMDD)

This is a five character field which identifies the data of sample collection where:

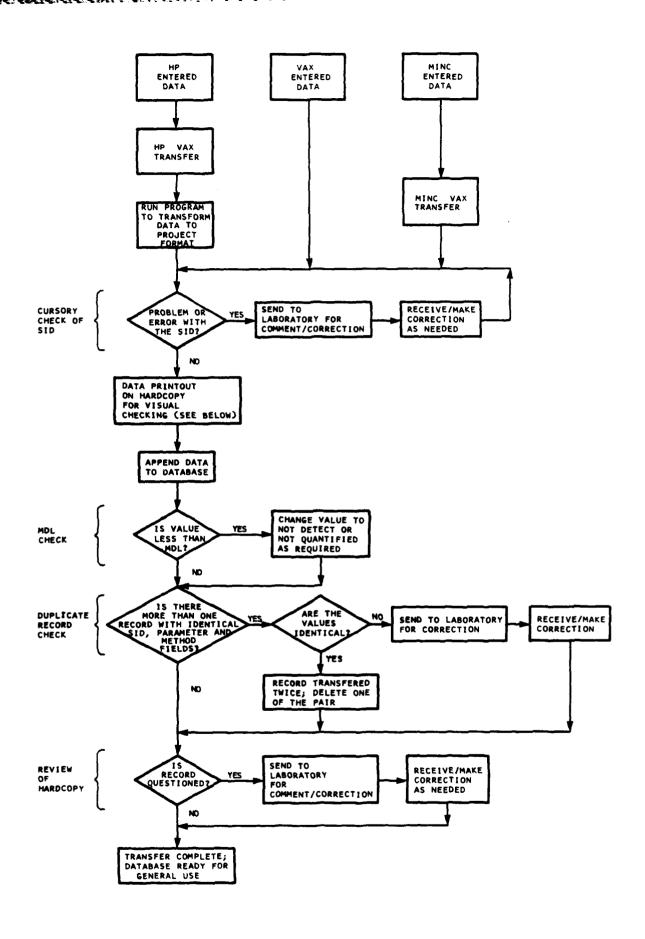
Y = Year A one digit number. (1981=1, 1982=2, 1983=3)

MM = Month A two digit number.

DD = Day A two digit number.

For composite samples, this is the day the sample was received.





Salar View

ZZZZZZZ

DATA FLOW THROUGH TRANSFER PROCESS FIGURE B. 2-2



especies acadeas incated increme accesses contin active accesse incidios addition actives

Accession (AAAAA)

This is a five character field which is typically reserved for the TPPAM. Five zeros are assigned if there is no accession code.

In addition to the sample identification codes, all parameters measured on this project were listed and numbered. In that some parameters were analyzed by more than one method, these parameters contain a single character "flavor" or "method" field, which follows the four character numeric parameter code. See Table B.2-3 for a listing of the parameter codes and methods.

Referring back to Figure B.2-1 as an example, the first record would be identified as Routine Water Quality Testing Program (Program = R), sampled at the Blend Tank Grab Tap (Location = 0762), collected at 7 A.M. (Time = 07) on 16 March 1981 (Date = 10316) and analyzed for turbidity (Parameter Code = 0570). The analytical result is 2.3

DATA ENTRY

Data was entered on the three computers previously mentioned by laboratory analysts and plant operators. At least thirty data entry programs were written and maintained for routine data entry purposes. A system of procedures was developed to check data from each of the data entry programs before it was added to the project's main database.

Each of the three computers had its own basic type of data entry programs. The programs maintained on the MINC computer were written in FORTRAN IV and utilize the FMS-11 (Forms Management System) software supplied by Digital Equipment Corporation. The software design for each program is similar and based on an initial systems analysis of on-site laboratory and operations procedures as well as hardcopy data forms or logs. The programs differ from one another only slightly, to match the unique manner in which data is collected for a particular analysis or plant operation.

The off-site laboratory utilized its Hewlett-Packard (HP) computer with an interactive program, Sample Information Management System (SIMS). The program has the following features:

- Sample log-in
- Sample chain of custody
- Analysts and sample receiving alerts
- Results acquisition
- Report generation
- Data validation/quality assurance
- Laboratory accounting

SIMS created a separate data file for each sample entered. On a periodic basis, sample files with analyses complete and results entered were transmitted to the VAX 11/780.



Once a file had been transferrred to the VAX, it required further processing before the data could be stored in the project database. This processing was accomplished with a program Compract, written in VAX-11 FORTRAN-IV Plus (Supplied by Digital Equipment Corporation). This program had three functions of interest to this project:

- 1. Resolved errors which arose from incompatibilities between the HP3354 and DEC VAX 11/780 hardware and software.
- 2. Translated codes used by the HP for sample identification into analogous codes on the VAX.
- 3. Extracted the sample identification code (SID) from each sample e and combined it with the translated VAX parameter code and the au tical result and formated the new record according to project do base specifications.

In cases where data was entered in the VAX, the data was stored directly in databases.

DATA QUALITY CONTROL

MANAGER SCHOOL WASHING INSIN SECTION

All data entry routines followed a basic pattern of entry and checking; entry by analyst or operator, followed by printout, review and correction of typographical errors before data was made available for transfer to the main project database. In most cases the data printouts were also reviewed by the analyst's supervisor before it could be transferred.

The term "transfer" refers to the process of moving (storing) data from their original datafiles to the project's primary database. Figure B.2-2 provides a graphical overview of the "flow of data" through the transfer process. At the top of the figure are boxes representing datafiles from each of the three different computers. All files go through a cursory check by the database manager where inconsistances, usually in the SID, are detected and corrected if possible. Some records needed to be sent to the laboratory for corrections. A system of flags, not shown in the figure, was implemented to track the path of a record as it was processed through the system.

Those records passing the cursory check were stored in an intermediate database and a hardcopy report was produced containing all the records in the database. The staff review consisted of examining the printout for errors detected by the computer, and checking analytical results for values outside the normal range. Such cases were then brought to the attention of laboratory analysts for checking and verification, and, in some cases, previously undetected errors (typographical and otherwise) were corrected.

After the printout was reviewed and confirmed, the newly transferred records were appended to the project's primary database.

The principal software utilized for project data analysis activities was Data-trieve-32, supplied by Digital Equipment Corporation. Datatrieve-32 is a query language and report writing system that runs on the VAX and permits direct, easy access to databases. Datatrieve-32 lets the computer user look at data, modify it or sort it interactively at the terminal. It allows privileged users to add new records (analytical results or operations data) to the data base, delete old ones or modify existing records to maintain an accurate, current file. In addition to its capabilities for data base maintenance, Datatrieve-32 provided an extensive report generation facility for simple as well as complex data reporting. Datatrieve-32 was also linked with other languages such as FORTRAN so that advanced users were able to take advantage of the best features of each type of software in a combined programming environment.

Software for preparation of statistical summary reports was written in VAX-11 FORTRAN IV Plus and callable Datatrieve-32. Its modular design allowed it to take advantage of the best features of both programming languages. The tables presented in Appendix F through H were generated using this software.

RS/1 was an additional data management system which was extensively used for analysis. RS/1 was specifically designed to meet the information handling requirements of applied scientific computer users, and was supplied to JMM by Bolt, Beranek, and Newman. It is based on a highly user oriented, English-like command language which permits the inexperienced computer user to make two dimensional data tables, simple x-y and bar graphs, perform more sophisticated statistical analyses and create models of simple and complex systems. It also provided the advanced user with an integrated programming language, RPL (Reseach Programming Language) for development of specialized or unique procedures.

Most of the graphical requirements were met by a graphics program developed by JMM called Easyplot. The program is designed around a commercially available package of FORTRAN callable graphics subroutines. The plots available include time series plots, cumulative probability plots with linear or logarithmic dependent axes, as well as all combinations of linear and logarithmic x-y data plots. Linear regression lines are available for all plot types, and Kolmogorov-Smirnov goodness of fit statistics were available on cumulative probability plots. Additional plotting capabilities were provided through the two data management software packages, Datatrieve and RS/1, as previously discussed.

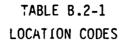
In addition to the software previously described, there were a number of specialized applications programs which were utilized for this project. These programs were either developed by JMM or acquired through outside sources and modified by JMM for specific applications. The following is a brief description of four such programs.

1. The Environmental Protection Agency program for estimation of costs for construction, operation and maintenance of water treatment facilities was obtained from the Municipal Environmental Research Lab in Cincinnati,



Ohio. This program was utilized as an aid in the estimation of costs for the 200 MGD Potomac Estuary Water Treatment Plant. (See Chapter 11)

- CHEMTRT is a chemical equilibrium model which calculates the changes in water quality brought about by advanced water or wastewater treatment processes. It has the capability of computing chemical equilibria involving acid/base, coordination, solubility, redox, and adsorption phenomenon. The output is provided in tabular format and reports printed are specified in the input data. The program requires detailed information on the influent water quality to the treatment facility together with changes to this water quality brought about by treatment steps. The program calculates the chemical equilibrium between the species in the influent, determining which species precipitate, are adsorbed, or participate in redox reactions. CHEMTRT is an adaption of the REDEQL2 program developed by Morgan and Morel at California Institute of Technology. CHEMTRT was used for estimation of chemical dosage requirements for corrosion control in the full scale 200 MGD plant.
- 3. TOWER is a program for design of air strippers to remove volatile organic compounds from water. The program estimates alternative packed tower design criteria for selected influent and effluent water quality characteristics and provides cost estimates for the selected design. The program was used in the air stripping evaluation described in Chapter 10 and Appendix I.
- 4. The Homogenous Surface Diffusion Model (HSDM) was used to predict effluent concentration profiles for activated carbon adsorbers. A description of the HSDM and its assumptions are given in Appendix I, Section 3. The model was used to predict effluent breakthrough curves for various empty bed contact times and treatment objectives. From these predictions, usage rates were determined and used to estimate regeneration costs for alternative designs for the full-scale plant.



Location Code	Location Description
001 002	Travel Blank EEWTP-PAS Travel Blank PAS-EEWTP-PAS
002	CORPS sample
004	ERA sample
005	EPA sample
006	Off-Site Stationary Blank
015	Blue Plains Nitrified Effluent Intake
016	Blue Plains Nitrified Effluent Intake
017	Blue Plains Nitrified Effluent Intake
025	Potomac River Estuary Intake
026	Potomac River Estuary Intake
027 036	Potomac River Estuary Intake Combined Feed
040	Microscreen 1 (Potomac River Estuary) Influent
040	Microscreen 1 (Potomac River Estuary) Influent
044	Microscreen 1 (Potomac River Estuary) Effluent
046	Microscreen 1 (Potomac River Estuary) Effluent
050	Microscreen 2 (Blue Plains Nitrified Effluent) Influent Microscreen 2 (Blue Plains Nitrified Effluent) Influent
051	Microscreen 2 (Blue Plains Nitrified Effluent) Influent
054	Microscreen 2 (Blue Plains Nitrified Effluent) Effluent
056	Microscreen 2 (Blue Plains Nitrified Effluent) Effluent
066	Microscreens 1 & 2 Backwash
070	EEWTP Blend Tank Influent
071	EEWTP Blend Tank (Mixing Area) EEWTP Blend Tank Effluent
074 075	EEWTP Blend Tank Overflow Weir
075	EEWTP Blend Tank (Effluent)
111	Aeration Basin
116	Aeration Basin Effluent
121	Recycle from Backwash Holding Tank
122	Rapid Mix 1 Chemical Feed System
123	Rapid Mix 2 Chemical Feed System
126	Rapid Mix 2 Effluent
141	Flocculation Tank 1
142	Flocculation Tank 2
143	Flocculation Tank 3
144	Flocculation Tank 4 Flocculation Tank 1 Effluent
146 151	Sedimentation Tank
156	Sedimentation Tank Sedimentation Tank Effluent
157	Sedimentation Sludge Pump
162	Recarbonation Tank Chemical Feed System

TABLE B.2-1 (Continued) LOCATION CODES

Location Code	Location Description
164 165 166 172 174 210 220 221 224 226 227 228 230 231 234	Recarbonation Tank Effluent Recarbonation Tank Effluent Recarbonation Tank Effluent Intermediate Chlorine Addition Intermediate Ozone Addition Dual Media Filter Common Influent Dual Media Filter 1 Influent Dual Media Filter 1 Surface Dual Media Filter 1 Effluent Dual Media Filter 1 Effluent Dual Media Filter 1 Backwash Valve Dual Media Filter 1 Drain Valve Dual Media Filter 2 Influent Dual Media Filter 2 Surface Dual Media Filter 2
236 237 238 240 241 247 310 315 334 336 337 354 356 357	Dual Media Filter 2 Effluent Dual Media Filter 2 Backwash Valve Dual Media Filter 2 Drain Value Dual Media Filter Common Effluent Dual Media Filter Effluent Filtered Water Clear Well Blowdown Carbon Column Common Influent Carbon Column Common Downflow Influent Downflow GAC Column 1 Effluent Downflow GAC Column 1 Effluent Downflow GAC Column 2 Backwash Valve
374 376 377 390 391 395 411 412 413 414 415 421 422	Downflow GAC Column 3 Effluent Downflow GAC Column 3 Effluent Downflow GAC Column 3 Backwash Valve Final Carbon Column Effluent (Effluent Line) Final Carbon Column Effluent (GAC Clearwell) GAC Clearwell Effluent Ozone Injection, Pass 1 Ozone Injection, Pass 2 Ozone Injection, Pass 3 Ozone Injection, Pass 4 Ozone Injection, Pass 5 Ozone Injection, Pass 5 Ozone Injection, Pass 6 Ozone Injection, Pass 7



TABLE B.2-1 (Continued) LOCATION CODES

Location Code	Location Description
424 425 426 429 430 432 450	Ozone Injection, Pass 8 Ozone Injection, Passes 5-8 Ozone Contact Tank Effluent Ammonia Addition UV Contact Tank Final Chlorine Addition Chlorine Contact Tank
452 471	Post Ozone Final Chlorine Addition EEWTP Finished Water
475	EEWTP Finished Water Clearwell Pump
476 479	EEWTP Finished Water Clearwell Effluent EEWTP Finished Water
510	TDS Removal Common Influent
515 520	Dissolved Solids Removal Feed
520 521	Reverse Osmosis conditioned Influent Reverse Osmosis Stage 1, Cartridge 1 product
522	
523	
524 525	Reverse Osmosis Stage 1, Cartridge 4 product
525 526	Reverse Osomsis Stage 2, Cartridge 1 product Reverse Osmosis Stage 2, Cartridge 2 product
527	Reverse Osmosis Stage 3, Cartridge 1 product
528	Reverse Osmosis finished water
531 532	Reverse Osmosis Stage 1, Cartridge 1 reject Reverse Osmosis Stage 1, Cartridge 2 reject
533	Reverse Osmosis Stage 1, Cartridge 2 reject
534	Reverse Osmosis Stage 1. Cartridge 4 reject
535	Reverse Osmosis Stage 2, Cartridge 1 reject
536 537	Reverse Osmosis Stage 2, Cartridge 2 reject Reverse Osmosis Stage 3, Cartridge 1 reject
540	Electrodialysis
560	
620	
640 660	TDS Ozone Contact Tank
710	TDS Chlorine Contact Tank Sludge Holding Tank
711	Sludge Holding Tank
716	Sludge Holding Tank Pump
721 726	Backwash Holding Tank
726 731	Backwash Holding Tank Pump Sludge Thickener Tank
736	Sludge Thickener Tank Pump

TABLE B.2-1 (Continued) LOCATION CODES

Location	Location
Code	Description
916	Water Treatment Plant 1 Finished Water
917	Water Treatment Plant 1 Finished Water
926	Water Treatment Plant 2 Finished Water
927	Water Treatment Plant 2 Finished Water
936	Water Treatment Plant 3 Finished Water
937	Water Treatment Plant 3 Finished Water
940	Onsite Laboratory Milli-Q Water
941	Onsite Laboratory Deionized Water
950	Pilot Plant Floc Influent
956	Pilot Plant Floc Effluent
960	Pilot Plant Sed Basin Influent
966	Pilot Plant Sed Basin Effluent
971	Pilot Plant Column No. 1 Influent
972	Pilot Plant Column No. 2 Influent
973	Pilot Plant Column No. 3 Influent
974	Pilot Plant Column No. 4 Influent
975	Pilot Plant Column No. 5 Influent
976	Pilot Plant Column No. 6 Influent
977	Pilot Plant Column No. 7 Influent
978	Pilot Plant Filter Clear Well No. 1
981	Pilot Plant Column No. 1 Effluent
982	Pilot Plant Column No. 2 Effluent
983	Pilot Plant Column No. 3 Effluent
984	Pilot Plant Column No. 4 Effluent
985	Pilot Plant Column No. 5 Effluent
986	Pilot Plant Column No. 6 Effluent
987	Pilot Plant Column No. 7 Effluent
990	Pilot Air Stripper Influent
996	Pilot Air Stripper Effluent

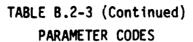
TABLE B.2-2 SAMPLER CODES

Sampler	Sampler
Code	Description
0	General
2	Grab
4	Continuous TOC
5	Probe
6	Flow Meter
7	Organics Concentrator
8	Virus Concentrator
9	Parasite Concentrator
Α	ACS Manning (No Preservative)
В	ACS Manning (Cyanide)
С	ACS EPA (TOC/TOX/THM)
Ð	ACS EPA (SOC)
Ε	ACS Solenoid
F	ACS EPA (Taste/Odor)
G	Manual Composite
Н	ACS Solenoid (Herbicides)
J	ACS Solenoid (Base/Neutrals)
K	ACS Solenoid (Acids)
L	ACS Solenoid (CLS)
M	ACS Solenoid (VOA)
N	ACS Solenoid (Asbestos/Tritium)
P	ACS Solenoid (Pesticides)
R	ACS Solenoid (Radiation)
T	Mechanical 1

CONSOLAR MARKER VARIOUS LINEARING MARKERS TRADUISM MARKERS, THINGERS MARKER TOWNERS TO THE SECOND

TABLE B.2-3 PARAMETER CODES

Parameter Code	Flavor	Parameter Description	Units
Code	L I GAOL	besci iption	Q.1.1.03
6		Volume	gallons
11		Headloss	Inches of water
17		Sequence	
18		Per Cent	X
21		Rotation speed	rpm
35		Depth	feet
62		Effective Size	mn
63		Uniformity Coefficient	
160	Α	Salmonella by 10 ml volume	MPN/100 m1
160	В	Salmonella by 100 ml volume	MPN/100 ml
160	C	Salmonella by 1000 ml volume	MPN/100 m1
163		Endotoxin	ng/ml
170		Standard Plate Count	colonies/ml
170	A	Standard Plate Count by 1 ml volume	colonies/ml
170	В	Standard Plate Count by 0.1 ml volume	colonies/ml 🚟
170	C	Standard Plate Count by 0.01 ml volume	colonies/ml
194	A	Total Coliform (confirmed) by 1000,100,10 ml vol.	MPN/100 m]
194	В	Total Coliform (confirmed) by 100,10,1 ml volume	MPN/100 m]
194	C	Total Coliform (confirmed) by 10,1,0.1 ml volume	MPN/100 m7
194	D	Total Coliform (confirmed) by 1,0.1,0.01 ml volume	MPN/100 m!
194	E	Total Coliform (confirmed) by 0.1,0.01,0.001 ml	MPN/100 m7
		volume	
194	F	Total Coliform (confirmed) by 0.01,0.001,0.0001 ml volume	MPN/100 ml
194	G	Total Coliform (confirmed) by 0.001,0.0001, 0.00001 ml volume	MPN/100 ml
196	Α	Total Coliform (completed) by 1000,100,10 ml vol.	MPN/100 m7
197	Â	Fecal Coliform (confirmed) by 1000,100,10 ml vol.	MPN/100 ml
197	B	Fecal Coliform (confirmed) by 100,10,1 ml volume	MPN/100 ml
197	Ď	Fecal Coliform (confirmed) by 1,0.1,0.01 ml volume	MPN/100 ml
197	Ē	Fecal Coliform (confirmed) by 0.1,0.01,0.001 ml volume	MPN/100 ml
197	F	Fecal Coliform (confirmed) by 0.01,0.001,0.0001 ml volume	MPN/100 ml
197	G	Fecal Coliform (confirmed) by 0.001,0.0001, 0.00001 ml volume	MPN/100 ml
370		MBAS	mg/L
373		UV Absorbance at 254 nm	Absorbance Units
373 3 9 0		Total Organic Halogen	ug/L-C1
392		Total Organic Carbon	



Parameter		Parameter	
Code	Flavor	Description	Units
392	A	Total Organic Carbon by DC80	mg/L-C
393		Total Organic Nitrogen	mg/L-N
412		Apparent Color	color units
420		Odor	TON
430		Taste	Taste Units
440		Temperature	deg. C
510		Alkalinity	mg/L-CaCO3
520		Electroconductivity	umho/cm
530		Hardness	
530	Α	Hardness by addition (Ca + Mg)	mg/L-CaCO3
540		pH	рĤ
545		Water Suitability Test	
550		Total Dissolved Solids (TDS) by drying	mg/L
550	Α	Total Dissolved Solids (TDS) by addition	mg/L
560		Total Suspended Solids (TSS)	mg/L
570		Turbidity	NTU
572		Nitrogen, Total Kjeldahl	mg/L-N
602		Calcium	mg/L
604		Potassium	mg/L
606		Magnesium	mg/L
608		Sodium	mg/L
610		Nitrogen, Ammonia	mg/L-N
620	Α	Silver by flame AAS	mg/L
620	В	Silver by furnace AAS	mg/L
622		Aluminum	mg/L
624		Arsenic	mg/L
626		Boron	mg/L
628		Barium	mg/L
630		Beryllium	mg/L
632	A	Cadmium by ICAP	mg/L
632	В	Cadmium by furnace AAS	mg/L
634	A	Cobalt by ICAP	mg/L
634	В	Cobalt by furnace AAS	mg/L
636	A	Chromium by ICAP	mg/L
636	В	Chromium by furnace AAS	mg/L
638 639	A	Copper by ICAP	mg/L
638 640	В	Copper by flame AAS	mg/L mg/L
640 642		Iron	
642 644	٨	Mercury	mg/L mg/L
644 644	A B	Lithium by ICAP Lithium by flame AAS	mg/L
648	U	Manganese	mg/L
650		Molybdenum	mg/L mg/L
ช่วบ		no i youchum	mg/ L

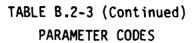


TABLE B.2-3 (Continued) PARAMETER CODES

Parameter		Parameter	
Code	Flavor	Description	Units
652		Nickel	mg/L
654		Lead	mg/L
656		Antimony	mg/L
658		Selenium	mg/L
660		Silica	mg/L
661		Tin	mg/L
662		Titanium	mg/L
664		Thallium	mg/L
666		Vanadium	mg/L
668	A B	Zinc by ICAP	mg/L
668 710	D	Zinc by flame AAS Chloride	mg/L mg/L
710 720		Fluoride	mg/L
730		Nitrogen, Nitrite + Nitrate	mg/L-N
740		Sulfate	mg/L
750		Bromide	mg/L
760		Cyanide, Total	mg/L
770		Iodide	mg/L
780		Ortho Phosphate	mg/L-P
786		Nitrate	mg/L-N
812		Total Chlorine	mg/L-C1
814 820		Free Chlorine Chlorine Remand	mg/L-C1
821		Chlorine Demand Chlorine Dose (Chlorine Demand Test)	mg/L-C1
822		Contact Time (Chlorine Demand Test)	minutes
823		Free Chlorine (Chlorine Demand Test)	mg/L-C1
824		Total Chlorine (Chlorine Demand Test)	mg/L-C1
825		pH (Chlorine Demand Test)	рЙ
826		Temperature (Chlorine Demand Test)	deg. C
827		Free Chlorine Criteria (Chlorine Demand Test)	mg/L-C1
917		Chrysotile Fiber Concentration	mf l
918		Amphibole Fiber Concentration	mf]
960 972		Dissolved Oxygen	mg/L
972 973		Gross Alpha Gross Alpha 2s Error	pCi/l
973 974		Gross Beta	pCi/l pCi/l
975		Gross Beta 2s Error	pCi/l
976		Tritium (Radiological)	pCi/l
977		Tritium 2s error	pCi/l
979		Strontium-90	pCi/l
980		Strontium-90 2s error	pCi/l
1231		Particles channel 1, 2.00-2.36 microns	
1232		Particles channel 2, 2.36-2.78 microns	

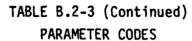
OF SCHOOL REPORTED TO SCHOOL TRANSPORT

garra meskessa ikastaka bennaba bendaba kakakak



Parameter		Parameter	
Code	Flavor	Description	Units
	Flavor		Units
1247		Particles channel 17, 28.26-33.34 microns	
1248		Particles channel 18, 33.34-39.34 microns	
1249		Particles channel 19, 39.34-46.42 microns	
1250		Particles channel 20, 46.42-54.78 microns	
1251		Particles channel 21, 54.78-64.64 microns	
1252		Particles channel 22, 64.64-76.28 microns	
1253 1254		Particles channel 23, 76.28-90.00 microns	
1254		Particles channel 24, 90.00 microns	
1261		24-Channel particles Y-intercept (alpha)	
1262		24-Channel particles slope (beta) 24-Channel particle counter mean	
1264		24-Channel particle sample dilution (1:x)	
1265		24-Channel particle sample volume	
2011	Α	Bromomethane by purge & trap GCMS	ug/L
2012	Ä	Chloromethane by purge & trap GCMS	ug/L ug/L
2013	A	Bromochloromethane by purge & trap GCMS	ug/L
2015	Α	Dichloromethane (Methylene chloride) by	49/ 5
		purge & trap GCMS	ug/L
2020	В	Trichlorofluoromethane by purge & trap GCMS	ug/L
2025	Α	Dichlorodifluoromethane by purge & trap GCMS	ug/L
2030	Α	Carbon tetrachloride by LLE ECD	ug/L
2030	В	Carbon tetrachloride by purge & trap GCMS	ug/L
2040	A	Chloroform by LLE ECD	ug/L
2040	В	Chloroform by purge & trap GCMS	ug/L
2041	A	Total Potential Chloroform by LLE ECD	ug/L
2045	A	Bromodichloromethane by LLE ECD	ug/L
2045	В	Bromodichloromethane by purge & trap GCMS	ug/L
2045	D	Bromodichloromethane by CLS GCMS	ug/L
2046	Α	Total Potential Dichlorobromomethane by LLE ECD	ug/L

Parameter		Parameter	
Code	Flavor	Description	Units
2050	A	Dibromochloromethane by LLE ECD	ug/L
2050	В	Dibromochloromethane by purge & trap GCMS	ug/L
2050	D	Dibromochloromethane by CLS GCMS	ug/L
2051	Α	Total Potential Dibromochloromethane by LLE ECD	ug/L
2055	Α	Bromoform by LLE ECD	ug/L
2055	В	Bromoform by purge & trap GCMS	ug/L
2055	D	Bromoform by CLS GCMS	ug/L
2056	A	Total Potential Bromoform by LLE ECD	ug/L
2060	A	Iodoform by purge & trap GCMS	ug/L
2065	A	Dichloroiodomethane by LLE ECD	ug/L
2065	В	Dichloroiodomethane by purge & trap GCMS	ug/L
2070 2071	A	Total THMs by LLE ECD 7-Day Total Potential THM by LLE ECD	ug/L
2071	A A	Chloroethane by purge & trap GCMS	ug/L ug/L
2085	Â	1.1-Dichloroethane by purge & trap GCMS	ug/L ug/L
2090	Â	1,2-Dichloroethane by purge & trap GCMS	ug/L
2095	Â	Hexachloroethane by purge & trap GCMS	ug/L
2095	Ď	Hexachloroethane by Base neut. LLE GCMS	ug/L
2095	Ē	Hexachloroethane by BNA LLE GCMS	ug/L
2095	F	Hexachloroethane by CLS GCMS	ug/L
2100	A	1,1,1-Trichloroethane by LLE ECD	ug/L
2100	В	1,1,1-Trichloroethane by purge & trap GCMS	ug/L
2105	В	1,1,2-Trichloroethane by purge & trap GCMS	ug/L
2105	D	1,1,2-Trichloroethane by CLS GCMS	ug/L
2110	A	1,1,2,2-Tetrachloroethane by LLE ECD	ug/L
2110	В	1,1,2,2-Tetrachloroethane by purge & trap GCMS	ug/L
2110	D	1,1,2,2-Tetrachloroethane by CLS GCMS	ug/L
2120	A	Trichloroethene by LLE ECD	ug/L
2120	В	Trichloroethene by purge & trap GCMS	ug/L
2120	D	Trichloroethene by CLS GCMS	ug/L
2125	A	Tetrachloroethene by LLE ECD	ug/L
2125 2125	В	Tetrachloroethene by purge & trap GCMS Tetrachloroethene by CLS GCMS	ug/L
2123	D A	Chloroethene (Vinyl chloride) by purge & trap GCMS	ug/L
2135	Ä	1,1-Dichloroethene by purge & trap GCMS	ug/L ug/L
2139	Â	cis-1,2-Dichloroethene by purge & trap GCMS	ug/L ug/L
2140	Â	trans-1,2-Dichloroethene by purge & trap GCMS	ug/L
2143	Ä	cis-1,2-Dichloropropene by Purge & trap GCMS	ug/L
2145	Ä	cis-1,3-Dichloropropene by purge & trap GCMS	ug/L
2150	Ä	Hexachlorobutadiene by purge & trap GCMS	ug/L
2150	Ď	Hexachlorobutadiene by Base neut. LLE GCMS	ug/L
2150	E	Hexachlorobutadiene by BNA LLE GCMS	ug/L
2150	F	Hexachlorobutadiene by CLS GCMS	ug/L



Parameter		Parameter	
Code	Flavor	Description	Units
2155		2 Chlanasthylyinylathan by numes 8 tuan CCMC	/1
2155 2155	A B	2-Chloroethylvinylether by purge & trap GCMS	ug/L
2155	Č	2-Chloroethylvinylether by Base neut. LLE GCMS 2-Chloroethylvinylether by BNA LLE GCMS	ug/L
2165	A		ug/L
2175	Ä	trans-1,3-Dichloropropene by purge & trap GCMS	ug/L
2175	Ĉ	1,2-Dichloropropane by purge & trap GCMS 1,2-Dichloropropane by CLS GCMS	ug/L
2176	В	1,2-Dibromo-3-chloropropane by purge & trap GCMS	ug/L ug/L
2180	Č	1,1'-0xybis(2-chloroethane) by Base neut. LLE GCMS	
2180	Ď	1,1'-0xybis(2-chloroethane) by BNA LLE GCMS	ug/L ug/L
2180	E	1,1'-0xybis(2-chloroethane) by CLS GCMS	ug/L ug/L
2185	Č	2,2'-0xybis(2-chloropropane) by	ug/L
L103	·	Base neut. LLE GCMS	ug/L
2185	D	2,2'-Oxybis(2-chloropropane) by BNA LLE GCMS	ug/L
2195	Ä	Alpha-BHC by LLE ECD	ug/L
2200	Ä	Beta-BHC by LLE ECD	ug/L
2205	Ä	Gamma-BHC by LLE ECD	ug/L
2210	Ä	Delta-BHC by LLE ECD	ug/L
2215	Ä	Kepone by LLE ECD	ug/L
2220	A	Toxaphene by LLE ECD	ug/L
2230	D	Hexachlorocyclopentadiene by Base neut. LLE GCMS	ug/L
2230	Ε	Hexachlorocyclopentadiene by BNA LLE GCMS	ug/L
2230	F	Hexachlorocyclopentadiene by CLS GCMS	ug/L
2235	A	Aldrin by LLE ECD	ug/L
2240	A	Dieldrin by LLE ECD	ug/L
2245	A	Chlordane by LLE ECD	ug/L
2250	A	Heptachlor by LLE ECD	ug/L
2255	A	Heptachlor epoxide by LLE ECD	ug/L
2260	Ą	Endrin by LLE ECD	ug/L
2265	Ą	Methoxychlor by LLE ECD	ug/L
2275	Ç	Fluoranthene by Base neut. LLE GCMS	ug/L
2275	D	Fluoranthene by BNA LLE GCMS	ug/L
2280	В	Benzo(b)fluoranthene by Base neut. LLE GCMS	ug/L
2280	C	Benzo(b)fluoranthene by BNA LLE GCMS	ug/L
2285	В	Benzo(k)fluoranthene by Base neut. LLE GCMS	ug/L
2285	C	Benzo(k)fluoranthene by BNA LLE GCMS	ug/L
2290	B	Benzo(g,h,i)perylene by Base neut. LLE GCMS	ug/L
2290 2295	C B	Benzo(g,h,i)perylene by BNA LLE GCMS	ug/L
	Č	Indeno(1,2,3-cd)pyrene by Base neut. LLE GCMS	ug/L
2295 2300	В	Indeno(1,2,3-cd)pyrene by BNA LLE GCMS Renzo(2)pyrene by Rase pout LLE GCMS	ug/L
2300	Č	Benzo(a)pyrene by Base neut. LLE GCMS Benzo(a)pyrene by BNA LLE GCMS	ug/L
2303	C	Pyrene by Base neut. LLE GCMS	ug/L ug/L
2303	D	Pyrene by BNA LLE GCMS	ug/L ug/L
2303	U	THE OF DIM LEE GOID	·9/ L



Sympto Supplies Yoursen I vincent Supplies

TABLE B.2-3 (Continued) PARAMETER CODES

Parameter Code	Flavor	Parameter Description	Units
2210		,	
2310	A	Benzene by purge & trap GCMS	ug/L
2315	A	Toluene by purge & trap GCMS	ug/L
2315	C A	Toluene by CLS GCMS	ug/L
2316 2316	Ĉ	4-Chloro-1-methylbenzene by purge & trap GCMS	ug/L
2325	A	4-Chloro-1-methylbenzene by CLS GCMS 1,2-Xylene by purge & trap GCMS	ug/L
2325	Ĉ	1,2-Xylene by CLS GCMS	ug/L
2330			ug/L
2330	A C	1,3-Xylene/1,4-Xylene by purge & trap GCMS 1,3-Xylene/1,4-Xylene by CLS GCMS	ug/L
2335			ug/L
2335	A	Ethylbenzene by purge & trap GCMS Ethylbenzene by CLS GCMS	ug/L
	C		ug/L
2340	A	Propylbenzene by purge & trap GCMS	ug/L
2340	C	Propylbenzene by CLS GCMS	ug/L
2345	A	Ethenylbenzene by purge & trap GCMS	ug/L
2345	Ç	Ethenylbenzene by CLS GCMS	ug/L
2355	В	2-Chloronaphthalene by purge & trap GCMS	ug/L
2355	C E	2-Chloronaphthalene by Base neut. LLE GCMS	ug/L
2355	F	2-Chloronaphthalene by BNA LLE GCMS	ug/L
2355 2360		2-Chloronaphthales by CLS GCMS	ug/L
	A	1-Chloronaphthalene by purge & trap GCMS	ug/L
2360	C	1-Chloronaphthalene by Base neut. LLE GCMS	ug/L
2360	D	1-Chloronaphthalene by BNA LLE GCMS	ug/L
2360	E	1-Chloronaphthalene by CLS GCMS	ug/L
2363	C	Bromobenzene by Base neut. LLE GCMS	ug/L
2363	ם	Bromobenzene by purge & trap GCMS	ug/L
2363	Ē	Bromobenzene by BNA LLE GCMS	ug/L
2363	F	Bromobenzene by CLS GCMS	ug/L
2365	A	Chlorobenzene by purge & trap GCMS	ug/L
2365	C	Chlorobenzene by CLS GCMS	ug/L
2370	A	1,2-Dichlorobenzene by purge & trap GCMS	ug/L
2370	D	1,2-Dichlorobenzene by Base neut. LLE GCMS	ug/L
2370	Ē	1,2-Dichlorobenzene by BNA LLE GCMS	ug/L
2370	F C	1,2-Dichlorobenzene by CLS GCMS	ug/L
2375		1,3-Dichlorobenzene by purge & trap GCMS	ug/L
2375	D	1,3-Dichlorobenzene by Base neut. LLE GCMS	ug/L
2375	E	1,3-Dichlorobenzene by BNA LLE GCMS	ug/L
2375	F	1,3-Dichlorobenzene by CLS GCMS	ug/L
2380	C	1,4-Dichlorobenzene by purge & trap GCMS	ug/L
2380	Ď	1,4-Dichlorobenzene by Base neut. LLE GCMS	ug/L
2380	Ē	1,4-Dichlorobenzene by BNA LLE GCMS	ug/L
2380	F	1,4-Dichlorobenzene by CLS GCMS	ug/L
2383	Α	1,3,5-Trichlorobenzene by purge & trap GCMS	ug/L

SYSTEM DESCRIPTION RECORDS RECORD STATES STATES RECORDS SOONS SOONS SECURIOR SOONS SOONS STATES

Parameter		Parameter	
Code	Flavor	Description	Units
2383	•	1,3,5-Trichlorbenzene by CLS GCMS	us /1
2384	C A	1,2,3-Trichlorobenzene by purge & trap GCMS	ug/L ug/L
2384	ĉ	1,2,3-Trichlorobenzene by CLS GCMS	ug/L ug/L
2385	B	1,2,4-Trichlorobenzene by purge & trap GCMS	ug/L
2385	Ď	1,2,4-Trichlorobenzene by Base neut. LLE GCMS	ug/L
2385	Ē	1,2,4-Trichlorobenzene by BNA LLE GCMS	ug/L
2385	F	1,2,4-Trichlorobenzene by CLS GCMS	ug/L
2386	В	1-Chloro-3-nitrobenzene by Base neut. LLE GCMS	ug/L
2386	C	1-Chloro-3-nitrobenzene by BNA LLE GCMS	ug/L
2387	В	Hexachlorobenzene by Base neut. LLE GCMS	ug/L
2387	D	Hexachlorobenzene by BNA LLE GCMS	ug/L
2387	Ε	Hexachlorobenzene by CLS GCMS	ug/L
2388	В	1-Chloro-2-nitrobenzene by Base neut. LLE GCMS	ug/L
2388	C	1-Chloro-2-nitrobenzene by BNA LLE GCMS	ug/L
2389	В	1-Chloro-4-nitrobenzene by Base neut. LLE GCMS	ug/L
2389	Ç	1-Chloro-4-nitrobenzene by BNA LLE GCMS	ug/L
2400	A	Acetone by purge & trap GCMS	ug/L
2410	Ą	2-Butanone by purge & trap GCMS	ug/L
2420	A	Tetrahydrofuran by purge & trap GCMS	ug/L
2450	C	Methylisoborneol by CLS GCMS	ug/L
2470	Ç	Geosmin by CLS GCMS	ug/L
2490	A	Napthalene by purge & trap GCMS	ug/L
2490	D	Napthalene by Base neut. LLE GCMS	ug/L
2490 2490	E F	Napthalene by BNA LLE GCMS	ug/L
2 49 0 2510	ם	Napthalene by CLS GCMS	ug/L
2510	E	Acenaphthylene by Base neut. LLE GCMS	ug/L
2520	D	Acenaphthylene by BNA LLE GCMS Acenaphthene by Base neut. LLE GCMS	ug/L
2520	Ē	Acenaphthene by BNA LLE GCMS	ug/L ug/L
2520	F	Acenaphthene by CLS GCMS	ug/L ug/L
2535	В	N-Nitrosodimethylamine by Base neut. LLE GCMS	ug/L
2535	Č	N-Nitrosodimethylamine by BNA LLE GCMS	ug/L
2540	č	Phenol by Acid LLE methyl GCMS	ug/L
2540	Ď	Pheno1 by BNA LLE GCMS	ug/L
2545	B	2-Chlorophenol by Acid LLE methyl GCMS	ug/L
2545	Ċ	2-Chlorophenol by BNA LLE GCMS	ug/L
2546	B	3-Chlorophenol by Acid LLE methyl GCMS	ug/L
2546	C	3-Chlorophenol by BNA LLE GCMS	ug/L
2547	В	4-Chlorophenol by Acid LLE methyl GCMS	ug/L
2547	C	4-Chlorophenol by BNA LLE GCMS	ug/L
2550	C	N-Nitrosodipropylamine by Base neut. LLE GCMS	ug/L
2550	D	N-Nitrosodipropylamine by BNA LLE GCMS	ug/L
2555	С	Nitrobenzene by Base neut. LLE GCMS	ug/L
2555	D	Nitrobenzene by BNA LLE GCMS	ug/L

Parameter		Parameter	
Code	Flavor	Description	Units
2560	C	Isophorone by Base neut. LLE GCMS	ug/L
2560	D	Isophorone by BNA LLE GCMS	ug/L
2565	В	4-Nitrophenol by Acid LLE methyl GCMS	ug/L
2565	C	4-Nitrophenol by BNA LLE GCMS	ug/L
2570	В	2-Nitrophenol by Acid LLE methyl GCMS	ug/L
2570	C	2-Nitrophenol by BNA LLE GCMS	ug/L
2575	В	2,4-Dimethylphenol by Acid LLE methyl GCMS	ug/L
2575	C	2,4-Dimethylphenol by BNA LLE GCMS	ug/L
2580	С	1,1'-(Methylenebis(oxy))-bis-2-chloroethane by	44
	_	Base neut. LLE GCMS	ug/L
2580	D	1,1'-(Methylenebis(oxy))-bis-2-chloroethane by	41
	_	BNA LLE GCMS	ug/L
2585	В	2,4-Dichlorophenol by Acid LLE methyl GCMS	ug/L
2585	C	2,4-Dichlorophenol by BNA LLE GCMS	ug/L
2590	В	2-Chloro-3-methylphenol by Acid LLE Methyl GCMS	ug/L
2590 2594	C	2-Chloro-3-methylphenol by BNA LLE GCMS	ug/L
2594	В	4-Chloro-3-methylphenol by Acid LLE methyl GCMS	ug/L
2594 2505	C	4-Chloro-3-methylphenol by BNA LLE GCMS	ug/L
2595 2505	В	2,4,6-Trichlorophenol by Acid LLE methyl GCMS	ug/L
2595 2506	C	2,4,6-Trichlorophenol by BNA LLE GCMS	ug/L
2596 2506	В	2,4,5-Trichlorophenol by Acid LLE methyl GCMS	ug/L
2596 2597	C	2,4,5-Trichlorophenol by BNA LLE GCMS	ug/L
2597 2597	B C	2,3,5-Trichlorophenol by Acid LLE methyl GCMS	ug/L
2598	В	2,3,5-Trichlorophenol by BNA LLE GCMS 2,3,6-Trichlorophenol by Acid LLE methyl GCMS	ug/L ug/L
2598	Č	2,3,6-Trichlorophenol by BNA LLE GCMS	ug/L ug/L
2600	В	Dimethylphthalate by Base neut. LLE GCMS	ug/L ug/L
2600	Č	Dimethylphthalate by BNA LLE GCMS	ug/L
2601	B	Diethylphthalate by Base neut. LLE GCMS	ug/L
2601	Ď	Diethylphthalate by BNA LLE GCMS	ug/L
2605	В	2,4-Dinitrophenol by Acid LLE methyl GCMS	ug/L
2605	Č	2,4-Dinitrophenol by BNA LLE GCMS	ug/L
2612	B	2-Methyl-4,6-dinitrophenol by Acid LLE methyl GCMS	
2612	Č	2-Methyl-4,6-dinitrophenol by BNA LLE GCMS	ug/L
2615	B	1-Methyl-2,6-Dinitrobenzene by Base neut. LLE GCMS	
2615	Č	1-Methy1-2,6-Dinitrobenzene by BNA LLE GCMS	ug/L
2620	В	1-Methyl-2,4-dinitrobenzene by Base neut. LLE GCMS	
2620	С	1-Methyl-2,4-dinitrobenzene by BNA LLE GCMS	ug/L
2625	C	Fluorene by Base neut. LLE GCMS	ug/L
2625	D	Fluorene by BNA LLE GCMS	ug/L
2625	Ε	Fluorene by CLS GCMS	ug/L
2630	C	1-Chloro-4-phenoxybenzene by Base neut. LLE GCMS	ug/L
2630	D	1-Chloro-4-phenoxybenzene by BNA LLE GCMS	ug/L
2630	Ε	1-Chloro-4-phenoxybenzene by CLS GCMS	ug/L

Parameter		Parameter	
Code	Flavor	Description	Units
2631	C	1-Bromo-4-phenoxybenzene by Base neut. LLE GCMS	ug/L
2631	D	1-Bromo-4-phenoxybenzene by BNA LLE GCMS	ug/L
2631	Ε	1-Bromo-4-phenoxybenzene by CLS GCMS	ug/L
2645	C	1,2-Diphenylhydrazine/Azobenzene by	
		Base neut. LLE GCMS	ug/L
2645	D	1,2-Diphenylhydrazine/Azobenzene by BNA LLE GCMS	ug/L
2645	E	1,2-Diphenylhydrazine/Azobenzene by CLS GCMS	ug/L
2650	В	Pentachlorophenol by Acid LLE methyl GCMS	ug/L
2650	C	Pentachlorophenol by BNA LLE GCMS	ug/L
2655	C	Phenanthrene by Base neut. LLE GCMS	ug/L
2655	D	Phenanthrene by BNA LLE GCMS	ug/L
2655	Ε	Phenanthrene by CLS GCMS	ug/L
2660	С	Anthracene by Base neut. LLE GCMS	ug/L
2660	D	Anthracene by BNA LLE GCMS	ug/L
2660	E	Anthracene by CLS GCMS	ug/L
2665	В	Di-n-Butylphthalate by Base neut. LLE GCMS	ug/L
2665	C	Di-n-Butylphthalate by BNA LLE GCMS	ug/L
2670	В	Benzidine by Base neut. LLE GCMS	ug/L
2670	C	Benzidine by BNA LLE GCMS	ug/L
2675	В	Benzylbutylphthalate by Base neut. LLE GCMS	ug/L
2675	C	Benzylbutylphthalate by BNA LLE GCMS	ug/L
2680	В	Bis(2-ethylhexyl)phthalate by Base neut. LLE GCMS	ug/L
2680	D	Bis(2-ethylhexyl)phthalate by BNA LLE GCMS	ug/L
2681 2601	B C	Diisobutylphthalate by Base neut. LLE GCMS	ug/L
2681 2682	В	Diisobutylphthalate by BNA LLE GCMS	ug/L
2682	C	Dicyclohexylphthalate by Base neut. LLE GCMS Dicyclohexylphthalate by BNA LLE GCMS	ug/L
2683	В	Diphenylphthalate by Base neut. LLE GCMS	ug/L ug/L
2683	C	Diphenylphthalate by BNA LLE GCMS	ug/L ug/L
2685	В	Chrysene by Base neut. LLE GCMS	ug/L ug/L
2685	Č	Chrysene by BNA LLE GCMS	ug/L
2690	В	Benzo(a)anthracene by Base neut. LLE GCMS	ug/L
2690	Č	Benzo(a)anthracene by BNA LLE GCMS	ug/L
2695	B	3,3'-Dichlorobenzidine by Base neut. LLE GCMS	ug/L
2695	Č	3,3'-Dichlorobenzidine by BNA LLE GCMS	ug/L
2700	B	Dioctylphthalate by Base neut. LLE GCMS	ug/L
2700	Č	Dioctylphthalate by BNA LLE GCMS	ug/L
2710	Ä	Arochlor 1016 by LLE ECD	ug/L
2715	Ä	Arochlor 1221 by LLE ECD	ug/L
2720	Ä	Arochior 1232 by LLE ECD	ug/L
2725	Ä	Arochlor 1242 by LLE ECD	ug/L
2730	A	Arochlor 1248 by LLE ECD	ug/L
2735	A	Arochior 1254 by LLE ECD	ug/L
		-	-



Parameter Code	Flavor	Parameter Description	Units
0000		5550. 155.01.	0
2740	Α	Arochlor 1260 by LLE ECD	ug/L
2750	C	2,4-D by LLE Methyl ECD	ug/L
2755	C C C	2,4,5-T by LLE Methyl ECD	ug/L
2760	C	2,4,5-TP by LLE Methyl ECD	ug/L
2765	Α	4,4'-DDE by LLE ECD	ug/L
2770	Α	4,4'-DDD by LLE ECD	∂g/L
2775	Α	4,4'-DDT by LLE ECD	ug/L
2780	Α	Endosulfan I by LLE ECD	ug/L
2785	Α	Endosulfan II by LLE ECD	ug/L
2790	Α	Endosulfan sulfate by LLE ECD	ug/L
2795	В	Dibenzo(a,h)anthracene by Base neut. LLE GCMS	ug/L
2795	C	Dibenzo(a,h)anthracene by BNA LLE GCMS	ug/L
2800	Α	1,2-Dibromoethane by purge & trap GCMS	ug/L
2800	В	1,2-Dibromoethane by CLS GCMS	ug/l
2800	C	1,2-Dibromoethane by CLS GCMS	ug/L
2805	В	Atrazine by Base neut. LLE GCMS	ug/L
2805	C	Atrazine by BNA LLE GCMS	ug/L
2810	В	Dioxin by Base neut. LLE GCMS	ug/L
2810	С	Dioxin by BNA LLE GCMS	ug/L
2815	В	Tricresolphosphate by Base neut. LLE GCMS	ug/L
2815	С	Tricresolphosphate by BNA LLE GCMS	ug/L
3084	C	N-Nitrosodiphenylamine by Base neut. LLE GCMS	ug/L
3084	D	N-Nitrosodiphenylamine by BNA LLE GCMS	ug/L
3107	Α	Dichloroacetonitrile by pentane ECD	ug/L
3108	Α	Bromochloroacetonitrile by pentane ECD	ug/L
3109	Α	Dibromoacetonitrile by pentane ECD	ug/L
8000		Chemicals (doses) 8000-8999	
8200		Alum	mg/L
8268		Hercofloc 1018	mg/L
8278		Betz 1160P	mg/L
8280		Calgon 233	mg/L
8500		Chlorine	mg/L
8550		Potassium permanganate	mg/L
8700		Lime	mg/L
8750		Caustic (Sodium Hydroxide)	mg/L
8755		Ammonia	mg/L
8800		Carbon Dioxide	mg/L

SECTION 3

Parameter Estimation for Partially Sensored Data: Evaluation of the EM Algorithm for an Independent Normal Process

prepared for

J.M. Montgomery Engineers 5020 Overlook Avenue, S.W. Washington, D.C. 20032

by

Eric F. Wood Consulting Hydrologist 227 Valley Road Princeton, NJ 08540

February, 1982

1. Introduction and Recommendations

This report addresses the problem of parameter estimation for probability density functions when the sampled data has been sensored. In the present context, sensored data represent water quality observations whose values lie below the level of detection of an analytical procedure.

The next section proposes an interative algorithm by which the parameters can be estimated, Section 3 presents results when the algorithm was used to estimate the parameters from a Normal probability density function. The results indicate that the algorithm gives results whose errors are equivalent to those expected if the parameters were being estimated from the complete data set.

In summary, I recommend that this algorithm be used by J.M. Montgomery Engineering in estimating the distribution parameters when sensored data is encountered. Furthermore, I recommend that J.M. Montgomery Engineering engineers carry out further statistical experiments to evaluate the algorithms over a wider range of situations than those covered in Section 3.

2. Iterative EM Algorithm

The EM algorithm is based upon the paper Dempster et. al., "Maximum Likelihood from Incomplete Date Via the

EM algorithm", J. Royal Statistical Society, B, 39, No. 1, pp. 1-22, 1977. The algorithm is iterative in nature and consists of two steps. Step 1 is to calculate the expected value of sufficient statistics for the sensored portion of the data set while step 2 is a maximization of the parameter estimates (as in maximum likelihood). Thus, EM comes from expectation-maximization.

One starts out with some initial expected value of the sensored data and uses this to maximize the likelihood of the parameter estimate over the sensored and unsensored data. The next step uses these parameter values to update the expected value of the sensored data. The algorithm iterates until convergence (usually 5 to 25 iterations depending upon the fraction of sensored data and the sample size).

2.1 Algorithm for an iid Normal process

Assume a sample size n where n_1 data were unsensored (x > D, D the detection level) and n_2 data are sensored (x < D). You want to calculate the mean and standard deviation for the underlying model.

Step 1: Calculate the partial sample statistics for the
 unsensored data (x > D)

mean =
$$n_1 = \frac{1}{n_1} \sum x_i$$
 $x_i > D$

variance =
$$s_1^2 = \frac{1}{(n_1-1)} \sum_{i=1}^{n} (x_i - M_1)^2$$

second moment
$$E[x^2|x>D] = S_1^2 + M_1^2$$

Step 2: Expectation Step

Step 2a: (for iteration 1) Initialize the first two moments of the sensored data. This can be done either by just setting them to reasonable values or by getting a fairly good estimate as set out in section 2.2.

The initial estimate $F(x = D) = n_2/n$, (F(x = D) being)the cumulative distribution function evaluated at x = D: $\int_{-\infty}^{D} f(x) dx$.

Go to Step 3.

Step 2b: (for iteration 2 onwards) Calculate the statistics $E[x|x \le D]$ and $E[x^2|x \le D]$ using the mean, m, and variance, S^2 , from Step 3 of the previous iteration. The equations for these are given in the Appendix.

Step 3: Maximizing Step

The population statistics (in our example the mean and variance) are estimated using

mean
$$\equiv M = M_2 \cdot F(x = D) + M_1 \cdot \{1 - F(x = D)\}$$

variance
$$E S^2 = E[x^2 | x \le D] \cdot F(x = D) + E[x^2 | x > D]$$

•
$$\{1 - F(x = D)\} - M^2$$

Steps 2b and 3 are repeated until the estimates converge. Limited experience indicates that if F(x = D) = .05 that five iterations should be sufficient. For the test results of section 3, F(x = D) = .15 and 20 iterations were more than sufficient.

2.2 Initializing M_2 and S_2^2

One could initialize M_2 and $E[x^2|x < D]$ by assuming that the pdf below the level of detection follows the exponential distribution

i.e.
$$f(x|x < D) = ae^{a(x-D)}$$
 for $x < D$

Then the 1st moment is
$$D - \frac{1}{a}$$
 (1)

2nd moment is
$$D^2 - \frac{2D}{a} + \frac{2}{a^2}$$
 (2)

and the variance is 1/a²

To evaluate the 1st and 2nd moments, we must estimate a (see Figure 1). One simple way is to set all x < D at some arbitrary value between D and O and calculate the whole sample mean and variance. Use this to estimate:

$$a = \frac{1}{\sqrt{2\pi}} \frac{1}{S} \exp \left[-\left(\frac{D-M}{\sqrt{2}S} \right)^2 \right]$$
 (3)

Use (1) and (2) with (3) for the initial estimates of $E[x|x \le D]$ and $E[x^2|x \le D]$ for the EM algorithm

3. Test Results

The EM algorithm was tested by generating a random sample of 500 distributed N(0,1) using an IMSL library subroutine (GGNML).

In analyzing the algorithm's performance, one must consider the uncertainty in the sample statistics

that can arise within the sample. Tables 1 through 4 report upon these sample statistics for 20 repititions of the random sample of 500.

The EM algorithm, to work well, should estimate well the following three statistics: $E[x|x \le D]$, $E[x^2|x \le D]$ and F(x = D) where x is the random variable and D the detention limit.

For a N(0,1) distribution, the exact values when D = -1.00 of these three statistics are:

$$E[x|x < D] = -1.525$$

$$E[x^{2}|x \le D] = 2.525$$

$$F[x = D] = .1587$$

The other statistic of importance, in so much that its uncertainty affects the results, is the expected value of the concentrations that are greater than the level of detection (E[x|x>D]). Its value for a N(0,1) process is .2876.

Table 1 gives the results for $E[x|x \le D]$ and E[x|x > D]. Columns 1 and 2 present the estimates when calculated from the sample mean and standard deviation based upon the full 500 samples. In columns 3 and 4, the same statistics are estimated using partial samples: that part less than D for column 3 and that part of the sample greater than D for column 4. Finally the result from the EM algorithm is given in column 5. Summary statistics



		•		
$E[x x \leq D]$	$E[x x \ge D]$	$E[x x \leq D]$	$E[x x \ge D]$	$E[x x \leq D]$
Calculated us-	calculated us-	estimated from	estimated from	calculated using
ing the sample	ing the sample	the partial	the partial	the EM algorithm
mean and std.	mean and std.	sample	sample	
d ev.	dev.	_		ļ
1.	2.	3.	4.	5.
- 1. 518	0.276	-1.522	0.274	-1.515
-1.551	0.328	- 1. 574	0.324	-1.559
-1.545	0.303	-1.514	0.296	-1.553
-1.533	0.308	-1.504	0.303	-1.559
- 1. 511	0.302	-1.469	0.328	-1.506
-1.510	0.263	-1.511	0.259	-1.506
-1.473	0.233	-1.500	0.234	-1.470
-1.433	0.227	-1.420	0.241	-1.476
-1.5J5	0.270	-1.522	0. 25 2	-1.517
-1.483	0.263	-1.496	0.275	-1.511
-1.5)2	0. 307	- 1. 530	0.268	- 1. 554
-1.498	0.300	-1.517	0.299	-1.514
- 1. 439	0.311	-1.443	0.278	-1.501
-1.503	0.325	-1.512	0.318	-1.541
- 1. 497	0.305	-1.494	0.313	-1.512
-1.490	0.299	- 1. 449	0.288	-1.537
-1.487	0.325	-1.520	0.311	- 1, 535
-1.474	0.268	-1.455	0.258	-1.500
-1.509	0.242	-1.454	0.225	-1.528
-1.537	0.202	-1.513	0.207	- 1. 485
VARI ABL	E MEAN S	STD. DEV.		
1	-1.5049	0.0227		
2	0.2841	0.0351		
3	-1.4963	0.0371		
3 4	0.2776	0.0346		
5	-1.5220	0.0278	·	

over the 20 repititions are also given and show that an average, the EM algorithm's estimate is closest to the true value.

Table 2 presents the results for the second moment statistics. Column 1 is the calculated value using the mean and standard deviation based upon the whole sample. Column 2, $E[x^2|x < D]$ is estimated from the partial sample where $x \le D$ and column 3 gives the EM algorithm result. The EM algorithm, on average, out performs the partial sample result.

Table 3 presents the results for the value of the cumulative at the detection level. Again, the value estimated by the EM algorithm out performs the estimates based on either the whole sample statistics or the partial sample.

Finally Table 4 presents the results for the distribution mean and standard deviation. Columns 1 and 2 give the complete sample estimates while columns 3 and 4 are the estimates using the EM algorithm. As shown in the summary for the 20 repititions, the EM algorithm, on average, estimated the true mean and standard deviation (0,1) better than the whole sample estimates.

The test results support the usefulness of the EM algorithm when sensored data are present. In fact, its performance is rather impressive.

Table 2

		•
calculated using	calculated using	calculated using
the sample mean	the partial sample	
and std. dev.	•	
_		
$E[x^2 x \leq D]$	$E[x^2 x \leq D]$	$E[x^2 x \leq D]$
- [m m -] D]		
1.	2.	3.
<u> </u>		J.
2 670	2.505	() 15 TH 4
2.478	2.506	2.471
2.687	2.680	2.676
2.589	2.550	2.566
2.607	2.428	2.593
2.572	2.346	2.662
2.428	2.494	2.412
2.326	2.431	2.306
2.267	2. 168	2.337
2.453	2.540	2.388
2.491	2.376	2.471
2.584	2.568	2.454
2.557	2.471	2.434
2.588	2.265	2.492
2.648	2.442	2.647
2.577	2.414	2.611
2.548	2.234	2.531
2.637	2.454	2.519
2.431	2.301	2.407
2.349	2.297	2. 27 š
2.184	2.520	2.212
	<u> </u>	
	MEAN STD. DEV.	
1 .	2.5010 0.13	33
2	2.4242 0.12	7 1
2 3	2.4848 0.13	54

Table 3

0.1572 0.1560 0.1568	
0. 1635 0. 1600 0. 1685 0. 1660 0. 1660 0. 1716 0. 1581 0. 1580 0. 1732 0. 1482 0. 1640 0. 1406 0. 1560 0. 1540 0. 1546 0. 1409 0. 1360 0. 1401 0. 1491 0. 1629 0. 1420 0. 1518 0. 1420 0. 1619 0. 1374 0. 1320 0. 1540 0. 1425 0. 1220 0. 1782 0. 1418 0. 1400 0. 1503 0. 1349 0. 1220 0. 1797 0. 1397 0. 1360 0. 1605 0. 1404 0. 1360 0. 1463 0. 1378 0. 1360 0. 1649 0. 1354 0. 1320 0. 1587 0. 1354 0. 1560 0. 1748	

VAFIABLE	MEAN	STD. DEV.
1	0.1488	0.0135
2	0.1465	0.0173
3	0.1591	0.0123

Table 4

Calculated mean using the comp- lete sample	Calculated std. dev. using the complete sample 2.	Calculated mean using the EM algorithm	Calculated std. dev. using the EM algorithm 4.
-0.0062	0.9879	-0.0067	0.9857
0.0204	1.0412	0.0069	1.0486
-0.0041	1.0267	-0.0209	1.0329
U.0173	1.0151	-0.0196	1.0412
. 0.0332	0.9894	0.0700	0.9929
-0.0135	0.9753	-0.0139	0.9698
-0.0023	0.9269	-0.0050	0.9213
-0.0277	0.9345	-0.0324	0.9311
0.0005	0.9727	-0.0339	0.9790
0.0408	0.9529	-0.0004	0.9804
0.0489	0.9811	-0.0554	1.0233
0.0446	0.9745	0.0266	0.9917
0.0678	0.9677	- 0. 05 27	1.0338
0.0693	0. 9885	0.0197	1.0275
0.0524	0. 9757	0.0457	0.9936
0.0521	0.9652	-0.0127	1.0133
0.0879	0.9700	0.0185	1.0185
0.0322	0.9372	~ 0. 0077	0.9628
-0.0374	0.9671	-0.0819	0.9816
-0.1205	0. 9827	-0.0546	0.9301

VARIABLE	MLAH	SID. DEV.
1	0,0178	0.0471
2	0.9766	0.0285
3	-0.0091	0.0362
tı.	0 9929	0 3376

Appendix A

Derivation of the 1st and 2nd Moments

of x Conditional Upon x < D

1st Moment

Given a probability density function, f(x) and a level of detection D, the expected value of $x \mid x \leq D$ is

$$E[x|x \le D] = \frac{1}{F_D} \int_{-\infty}^{D} x f(x) dx$$
 (A1)

where F_D is the cummulative distribution function evaluated at x = D. That is

$$F_{D} = \int_{-\infty}^{D} f(x) dx$$
 (A2)

Assuming f(x) is a Normal probability density function,
(Al) can be written as

$$E[x | x \le D] = \frac{1}{F_D} \int_{-\infty}^{D} \frac{x}{\sqrt{\pi}} \exp\left[-\left(\frac{x-u}{\sqrt{2(S)}}\right)^2\right] \frac{ds}{\sqrt{2(S)}}$$
(A3)

where u is the mean and S is the standard deviation.

Equation (A3) can be integrated using the following change in variables; let $T = \frac{x-u}{\sqrt{2}S}$ then $dT = dx/\sqrt{2}S$ and and $x = \sqrt{2}ST + u$. Substituting this into (1) results in

$$E[x|x \le D] = \frac{1}{F_D} \int_{-\infty}^{\frac{D-U}{\sqrt{2}S}} \frac{\sqrt{2}ST + u}{\sqrt{\pi}} \exp(-T^2) dT$$
(A4)

which can now be divided into two integrals. The first part is

$$\frac{\sqrt{2} \text{ S}}{F_{D}\sqrt{\pi}} \int_{-\infty}^{D-u} \text{T} \exp(-\text{T}^{2}) d\text{T} = -\frac{\text{S}}{F_{D}\sqrt{2\pi}} \exp\left[-\left(\frac{D-u}{\sqrt{2}\text{ S}}\right)^{2}\right]$$
(A5)

The second part is

$$\frac{u}{F_{D}\sqrt{\pi}} \int_{-\infty}^{\frac{D-u}{\sqrt{2} S}} \exp(-T^{2}) dT = \frac{u}{2F_{D}} \left[\operatorname{erf} \left(\frac{D-u}{\sqrt{2} S} \right) + 1 \right]$$
(A6)

The evaluation of the second part utilizes the error function which is defined as

$$\frac{2}{\sqrt{\pi}} \int \exp(-T^2) dT = \operatorname{erf}(T)$$

and

$$erf(-\infty) = -1$$

 $\boldsymbol{F}_{\boldsymbol{D}}$ can be evaluated in a similar manner as:

$$F_{D} = \frac{1}{2} \left\{ erfc \left[-\frac{(D-u)}{\sqrt{2} s} \right] \right\}$$
 (A7)

where erfc(·) is the complimentary error function. Combining (A5), (A6) and (A7) results in

$$\mathbb{E}\left[\mathbf{x}\,\middle|\,\mathbf{x}\leq\mathbf{D}\right] = \frac{\frac{\mathbf{u}}{2}\left[\operatorname{erf}\left(\frac{\mathbf{D}-\mathbf{u}}{\sqrt{2}\,\mathbf{S}}\right) + 1\right] - \frac{\mathbf{S}}{\sqrt{2\pi}}\exp\left[-\left(\frac{\mathbf{D}-\mathbf{u}}{\sqrt{2}\,\mathbf{S}}\right)^{2}\right]}{\frac{1}{2}\left[\operatorname{erfc}\left(-\frac{\mathbf{D}-\mathbf{u}}{\sqrt{2}\,\mathbf{S}}\right)\right]} \tag{A8}$$

2nd Moment

The 2nd moment is defined as

$$E[x^{2}|x \leq D] = \frac{1}{F_{D}} \int_{-\infty}^{D} x^{2}f(x) dx \qquad (A9)$$

which for the Normal distribution, and using the change in variables presented above, results in (A9) being written as

$$E[x^{2}|x \le D] = \frac{1}{F_{D}} \int_{-\infty}^{\frac{D-U}{\sqrt{2} S}} \frac{(2S^{2}T^{2} + 2\sqrt{2} SuT + u^{2})}{\sqrt{\pi}} \exp(-T^{2})$$
(A10)

Equation (Al0) can be represented by the sum of three integrals of which the first is

$$\frac{2 \text{ S}^2}{\text{F}_D \sqrt{\pi}} \int_{-\infty}^{D-u} \text{T}^2 \exp(-\text{T}^2) dT \qquad (A11)$$

the second one is

$$\frac{2\sqrt{2} \text{ Su}}{F_{D}\sqrt{\pi}} \int_{-\infty}^{\frac{D-u}{\sqrt{2} \text{ S}}} T \exp(-T^2) dT = \frac{\sqrt{2} \text{ uS}}{F_{D}\sqrt{\pi}} \exp\left[-\left(\frac{D-u}{\sqrt{2} \text{ S}}\right)^2\right]$$
(A12)

utilizing equation (A5) and the third one is

$$\frac{u^2}{F_D\sqrt{\pi}} \int_{-\infty}^{\frac{D-u}{\sqrt{2} S}} \exp(-T^2) dT = \frac{u^2}{2F_D} \left[\operatorname{erf} \left(\frac{D-u}{\sqrt{2} S} \right) + 1 \right]$$
(Al3)

utilizing equation (A6)

Equation (All) must be integrated by parts. Ignoring the constant $2S^2/F_D$ $\sqrt{\pi}$ for the moment, let

$$du = -2T \exp(-T^2)dT$$
 $u = \exp(T^2)$

$$v = -\frac{T}{2} \qquad dv = -\frac{dT}{2}$$

Then equation (All) can be rewritten as $uv - \int udv$, which is $-\frac{T}{2} \exp(-T^2) \begin{vmatrix} \frac{D-u}{\sqrt{2} S} \\ + \frac{1}{2} \end{vmatrix} \int_{-\infty}^{D-u} \exp(-T^2) dT$

The integral is $\frac{\sqrt{\pi}}{4} \cdot \text{erf}(T)$. Evaluating at the integral limits and including the constant $2S^2/F_D\sqrt{\pi}$ gives the result

$$-\frac{s^2}{F_D\sqrt{\pi}}\left(\frac{D-u}{\sqrt{2} s}\right) \exp\left[-\left(\frac{D-u}{\sqrt{2} s}\right)^2\right] + \frac{s^2}{2F_D} \left[\operatorname{erf}\left(\frac{D-u}{\sqrt{2} s}\right) + 1\right]$$
(A15)

(A14)

Combining (Al2), (Al3) and (Al5), and using (A7) for ${\bf F}_{{\bf D}}$ gives our desired result

$$E[x^{2}|x \leq D] = \frac{\frac{u^{2}+S^{2}}{2} \left[erf\left(\frac{D-u}{\sqrt{2} S}\right) + 1 \right] - \left[\frac{\sqrt{2} uS}{\sqrt{\pi}} + \frac{S^{2}}{\sqrt{\pi}} \left(\frac{D-u}{\sqrt{2} S}\right) \right] exp\left[-\left(\frac{D-u}{\sqrt{2} S}\right)^{2}\right]}{\frac{1}{2} \left[erfc\left(-\frac{D-u}{\sqrt{2} S}\right) \right]}$$

(A16)

The variance of $x \mid x \leq D$, $v(x \mid x \leq D)$ can be easily calculated from

$$v[x|x \le D] = E[x^2|x \le D] - E^2[x|x \le D]$$
 (A17)

Equations (A8), (A16) and (A17) were used in the EM algorithm.

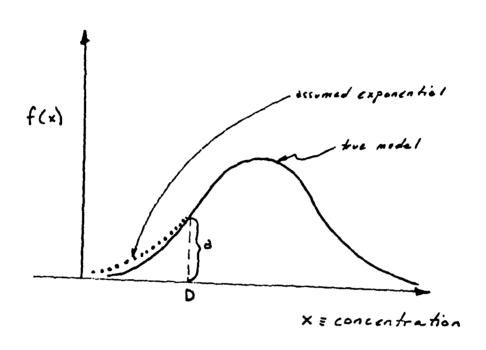


Figure 1

REFERENCES

- Blight, B. J. N., "Estimation From a Censored Sample for the Exponential Family," Biometrika, 57:389-395 (1970).
- Box, G. E. P., Hunter, W.G., and Hunter, J.S., Statistics for Experimenters, John Wiley and Sons, Inc., New York (1978).
- Cohen, A. C., Jr., "On the Solution of Estimating Equations for Truncated and Censored Samples from Normal Populations," <u>Biometrika</u>, 44:225-236.
- Dempster, A. P., Laird, N. M., and Rubin, D. B., "Maximum Likelihood from Incomplete Data via the EM Algorithm," J. Royal Statistical Society, B, 39(1):1-22 (1977).
- Grundy, P. M., "The Fitting of Grouped Truncated and Grouped Censored Normal Distributions," Biometrika, 39:252-259.
- Hald, A., "Maximum Likelihood Estimation of the Parameters of a Normal Distribution which is Truncated at a Known Point," Skandinavisk Aktuarietidskrift, 1949:119-134 (1949).
- Hald, A., Statistical Theory With Engineering Applications, John Wiley and Sons, Inc., New York (1952).
- Shapiro, S. S. and Wilk, M.B., "An analysis of variance test for normality (complete samples)," Biometrika, 52:591-611 (1965).
- Shapiro, S. S., Wilk, M. B., and Chen, H. J., "A Comparative Study of Various Tests for Normality," <u>Journal of the American Statistical Association</u>, 63:1343-1372 (1968).
- S* phens, M. A., "EDF Statistics for Goodness of Fit and Some Comparisons,"

 <u>Journal of the American Satistical Association</u>, 69:730-737 (1974).

APPENDIX C

DYNAMIC ESTUARY MODEL DESCRIPTION

INTRODUCTION

This appendix provides a more detailed discussion of the U.S. Environmental Protection Agency's (EPA) Dynamic Estuary Model (DEM) than that presented in Chapter 6, Section 1. The DEM was used in this study to predict levels of conservative water quality parameters in the Potomac River estuary under future low-flow conditions. The initial and boundary conditions used in this study, and described in the following sections, focus on the modeling of total dissolved solids (TDS). The application of DEM results for TDS in predicting levels of other water quality parameters is discussed in Chapter 6, Section 1.

Following a general description of the types of models available for investigations of hydraulics behavior and water quality of estuaries, this appendix describes the development of the EPA DEM, and the application of the DEM to estuary studies in the mid-Atlantic states.

TYPES OF MODELS

The two general types of models used for investigations of the hydraulics and water quality of estuaries are physical models and numerical models. Physical models are constructed based upon scaled relationships of forces governing behavior of the estuary between the model system and the prototype, or full-scale, system. These relationships are expressed in terms of dimensionless groups (e.g., Froude Number, Reynolds Number). In addition, the ratio of horizontal to vertical distances in the model system is often exagerated in order to maintain turbulent flow. Once constructed, physical estuary models undergo a painstaking process of calibration, where brass strips in the bottom of channels are individually adjusted (i.e., bent and/or twisted) so that model observations match those of the prototype.

The second type of model is a numerical model. These types of models were developed following the development of digital computers. This led to the cost-effective use of numerical methods for solving equations which describe the system. These equations describe the natural processes and governing forces controlling fluid flow, mass transport, and chemical and biological transformations of water quality parameters. Similar to physical models, numerical models are calibrated after construction by adjusting parameters in the governing equations. Numerical models can be used not only to predict hydrodynamic behavior and levels of conservative water quality parameters, but to predict levels of non-conservative parameters as well.



DEVELOPMENT OF THE DYNAMIC ESTUARY MODEL

The Dynamic Estuary Model is a numerical estuary model developed by Water Resources Engineers (Walnut Creek, California) for the U.S. Public Health Service, Division of Water Supply and Pollution Control in the mid-1960's. The DEM was then used by the U.S. Environmental Protection Agency (EPA) following that agency's formation in 1969. The DEM was first used for water quality studies in the San Francisco Bay and the Sacramento-San Juaquin Delta areas. The DEM was the first estuarine model to incorporate a "real time" solution of the hydrodynamic behavior of estuaries with tidal influences. The DEM consists of two components which can be run independently. hydrodynamic component is used to predict the movement of fluid in the estuary. The water quality component is used to predict mass transport and biological and/or chemical transformations of water quality parameters. The water quality component requires, as part of its input, the fluid flows in the estuary as predicted by the hydrodynamic component. However, this data may be input from magnetic tape generated from a previous run of the hydrologic component (EPA, 1979).

APPLICATIONS OF THE DYNAMIC ESTUARY MODEL IN THE MID-ATLANTIC AREA

POTOMAC RIVER ESTUARY

THE REPORTED BESIDES - PESSENCE - PROPERS CONTROL PROPOND CONTROL BONDON MAKKET

The DEM was extensively tested and modified by the EPA during several studies of water quality in the Potomac River Estuary. The goals of these studies were to refine the handling of nutrient cycles and to incorporate algal effects into the dissolved oxygen budget.

A recent summary of the performance of the DEM by Ambrose and Roesch (1982) indicated that the DEM performed very well in predicting hydrodynamics and mass transport in the Potomac estuary. Their study evaluated the predictive ability of the model based on accuracy and systematic errors (i.e, comparison of average, relative error, slope and intercept of linear regression parameters between model predictions and observed values) as well as precision (i.e., comparison of standard error of estimate, coefficient of variation, correlation coefficient between model predictions and observed values). Their study looked at model predictions and observed Potomac estuary data in the following three areas:

- Hydrodynamics (e.g., tidal parameters)
- Mass Transport and Circulation (e.g., dye tests and salinity levels)
- Non-conservative water quality parameters (e.g. dissolved oxygen, ammonia nitrogen, nitrate nitrogen, total phosphate, and chlorophyll)

For the hydrodynamic parameters, the correlation coefficient between DEM predictions and observed estuary data, ranged between 0.98 and 1.00. This indicated good precision. For mass transport parameters, the correlation coefficient between DEM predictions and observed estuary data ranged between

0.84 and 1.00. Their conclusion was that the DEM was basically reliable when operated within the range of conditions for which it was calibrated and tested, and that the DEM captured overall trends nicely.

OTHER ESTUARIES

The DEM has been applied in water quality studies for both the Chesapeake Bay and the Delaware River Estuary (Ambrose and Roesch, 1982). The DEM was used in a water quality study of the upper Chesapeake Bay to determine allowable nutrient loadings from the Susquehanna River Basin in the Baltimore Metropolitan Area. The DEM was also used in water quality studies for the Delaware River Estuary similar to those described above for the Potomac River Estuary.

REPRESENTATION OF POTOMAC RIVER ESTUARY GEOMETRY

CHARACTERISTICS OF "LINK-NODE" NETWORK

The DEM is a one-dimensional numerical model. That is, concentration variations are allowed only along the channel, or in the x-direction. The concentrations of water quality parameters are assumed to be homogeneous across the channel, or the y-direction, and throughout the water column, or the z-direction. In order to handle the geometry of estuaries using this one-dimensional representation, the "link-node" system was developed. This allows the DEM to represent wide or narrow channels as well as embayments in a particular estuary. Figures C.0-1 through C.0-6 illustrate the link-node network developed to describe the geometry of the Potomac River Estuary.

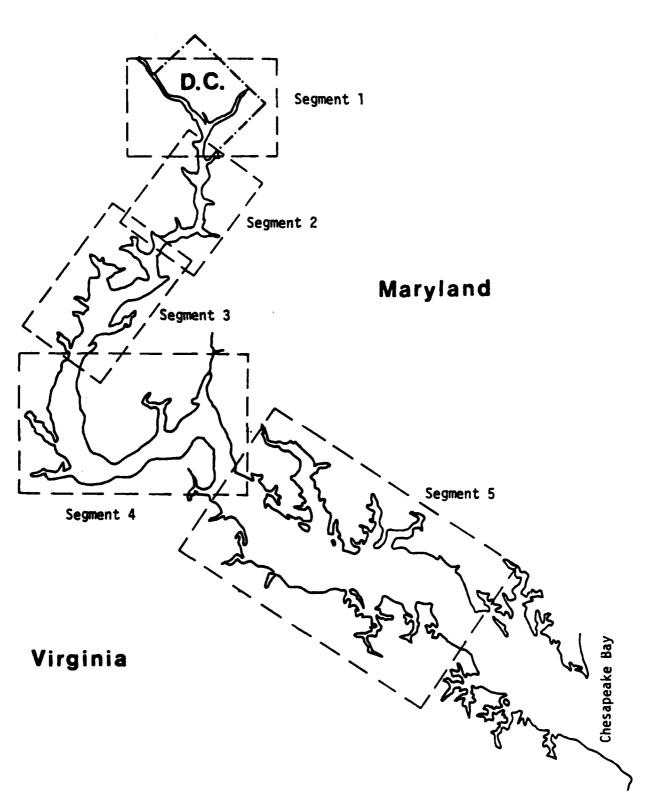
PARAMETERS DESCRIBING "LINK" CHARACTERISTICS

In the DEM, the "links" are treated as open channels for the transport of water and chemical mass between adjacent nodes. The following parameters are used in the model to describe the characteristics of links (EPA, 1979):

- Length the maximum length of a given link is governed by stability criteria which depend on the celerity (or speed) of a wave and the time step used.
- Width
- Cross Sectional Area this parameter will vary with tidal stage.
- Manning Roughness this parameter is used to "fine tune" the model for predicting the hydrodynamic behavior of the estuary.
- Water Velocity this parameter will vary with tidal stage.
- Hydraulic Radius this parameter will vary with tidal stage.

PARAMETERS DESCRIBING "NODE" CHARACTERISTICS

In the DEM, the nodes are equivalent to fluid reservoirs. Nodes may be joined by one or more links. The following parameters are used to describe the characteristics of nodes (EPA, 1979):



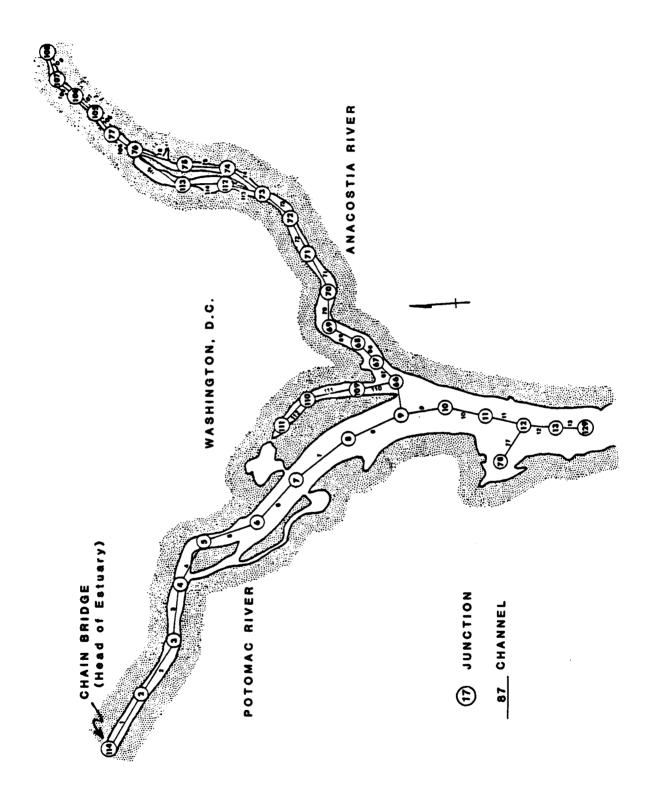
SEGMENTS COMPRISING
POTOMAC RIVER ESTUARY USED IN THE DEM
FIGURE C. 0-1

COURT , COURT , COURT

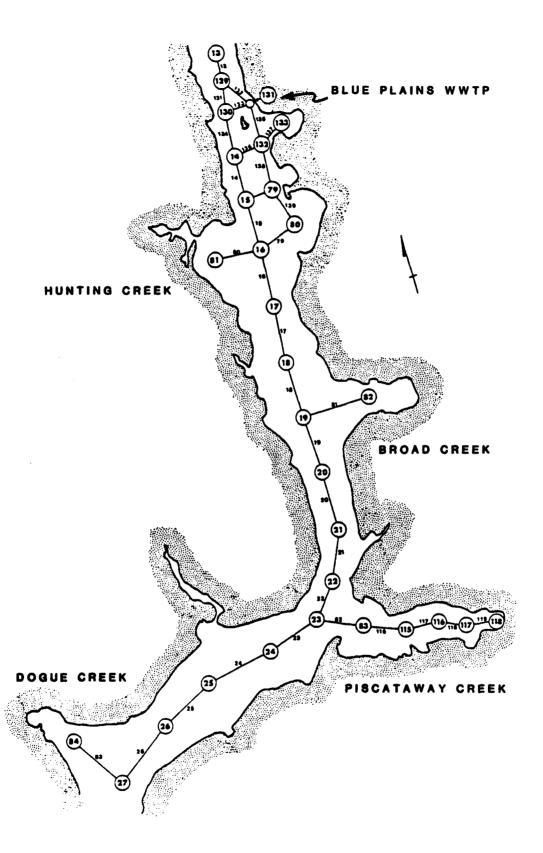


CONTRACTOR OF THE PROPERTY OF

Province Service Vectories

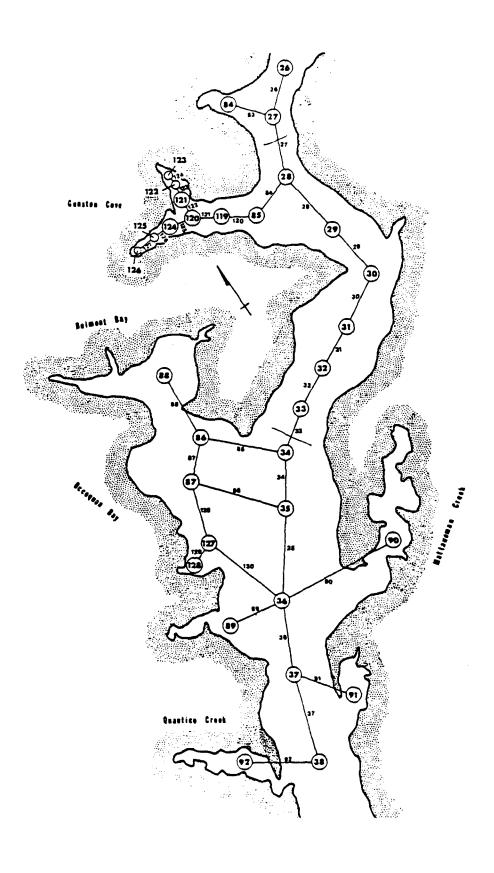


POTOMAC ESTUARY MODEL NETWORK SEGMENT 1 FIGURE C. 0-2

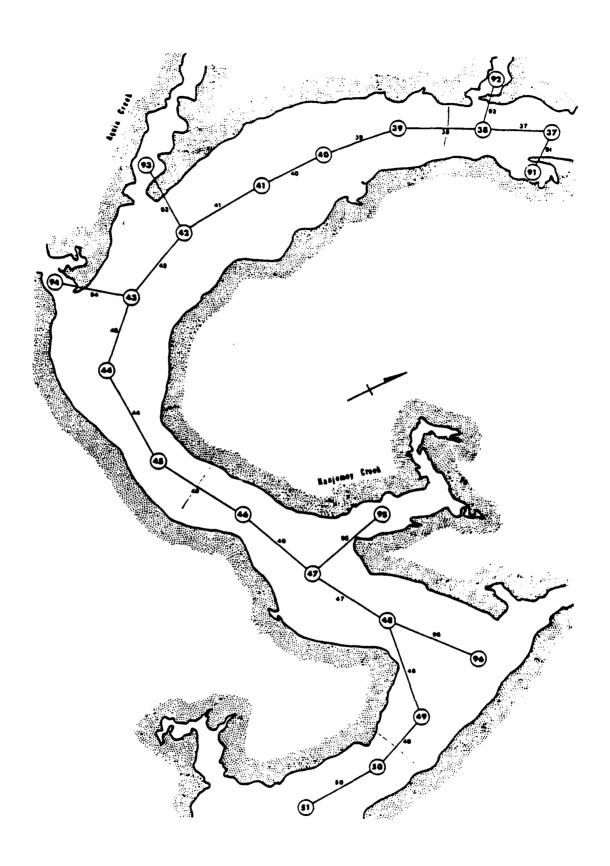


POTOMAC ESTUARY MODEL NETWORK SEGMENT 2 FIGURE C. 0-3

SON LEAGUEL LEAGUE PORTORY PROPERTY ASSESSED

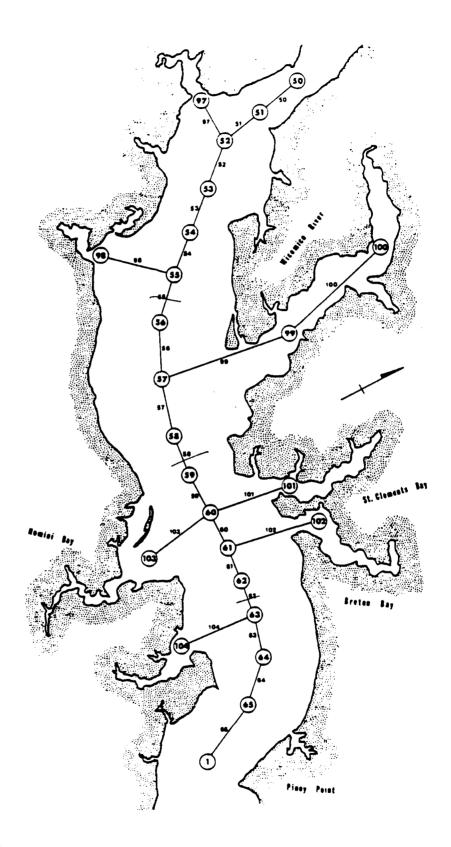


POTOMAC ESTUARY MODEL NETWORK SEGMENT 3 FIGURE C. 0 4



POTOMAC ESTUARY MODEL NETWORK SEGMENT 4 FIGURE C. 0-5

100 Kenton



POTOMAC ESTUARY MODEL NETWORK SEGMENT 5 FIGURE C. 0-6

- Surface Area
- Volume
- Head (or water elevation above a specified datum)
- Accretion/Depletion of Water and/or Mass

HYDROLOGIC COMPONENT

GOVERNING EQUATIONS AND ASSUMPTIONS

The hydraulic component of DEM incorporates two governing differential equations describing the propagation of a long wave through a shallow water system (i.e., ebb and flow of the tide in estuaries). The equation of motion is used to describe the conservation of momentum, and the equation of continuity is used to describe the conservation of mass. This approach used in the DEM for solving for fluid motion in an estuary is based upon the following assumptions (EPA, 1979):

- The flow is one dimensional.
- Acceleration perpendicular to the x-axis is negligible.
- Coriolis and wind forces are negligible.
- Channels are rectangular with a uniform cross-sectional area and negligible slope.
- The tidal conditions (i.e. period and amplitude) at the seaward boundary are known.
- Wave length is greater than or equal to two times the channel depth.

NUMERICAL SOLUTION TECHNIQUE

The solution of the two governing differential equations described above is based on a finite difference approximation. The hydraulic component of the DEM uses a modified Runge-Kutta technique for numerical integration (EPA, 1979; Kreysig, 1979).

INITIAL AND BOUNDARY CONDITIONS

SO VERFORM INSPIRED HOUSENING HENDERS REFEREN TORKERS PREFEREN

The DEM was obtained from the EPA and was modified to handle an estuary water treatment plant as described below. The model was calibrated for the Potomac River estuary by the EPA.

The rationale for selection of the initial and boundary conditions used in this study was described in Chapter 6, Section 1. Table C.0-1 lists the fresh water inflows (i.e., boundary conditions) from the Potomac River into the head of the estuary during the period modeled in this study. The fresh water inflow from the Anacostia River was held constant at 20 cfs (13 MGD) throughout the period modeled in this study.

Although two alternative locations for the proposed full-scale estuary water treatment plant were investigated using the DEM, the withdrawal of estuary water in both cases was 200 MGD. As described in Chapter 6, Section 1, the water withdrawn from the Potomac River and Potomac River Estuary for

TABLE C.0-1
FRESHWATER INFLOW BOUNDARY CONDITIONS AT CHAIN BRIDGE

		JULY			ALEUST		SEI	SEPTENBURG	
day.	1930 obs. (cfs)	-Chain (cfs)	Bridge (MGD)	1930 obs. (cfs)	(cfs)	Bridge (MGD)	1930 obs. (cfs)	c-Chain (cfs)	Bridge (MGD)
	•	550.	355.	602.	152.	98	. 599	215.	139.
~		550.	355.	594.	14.	93.	594.	144.	93.
~	•	550.	355.	570.	120.	п.	.599	215.	139.
÷	t	550.	355.	. 165	<u> </u>	93.	654.	707	132.
\$	•	\$50.	355.	621.	171.	110.	594.	14.	93.
9	•	550.	355.	632.	182.	117.	578.	128.	83.
7	,	550.	355.	594.	14.	93.	562.	112.	12.
20	١	550.	355.	654.	707	132.	. 946	96.	.79
6	,	550.	355.	.969	248.	160.	578.	128.	83.
2	,	550.	355.	610.	160.	103.	752.	307.	195.
=	ı	550.	355.	610.	160.	103.	1020.	570.	368.
71	t	550.	355.	610.	160.	103.	736.	. 982	185.
=	•	550.	355.	578.	128.	83.	632.	182.	117.
=	•	550.	355.	562.	112.	12.	65.1.	504·	132.
15	1	550.	355.	. 295	112.	12.	837.	382.	546.
91	•	550.	355.	516.	.93	1 3.	784.	334.	.512
13	ŧ	550.	355.	495.	45.	29.	832.	382.	746.
<u>2</u>	•	550.	355.	-181	31.	20.	632.	382.	746.
19	,	550.	355.	554.	104.	67.	736.	786.	185.
70	934.	484.	312.	594.	144.	93.	.929	. 777	146.
17	.088	130.	. 111.	546.	96.	. 62.	. 179	171.	-01
걲	768.	318.	.505	50.)	59.	38.	. 709	157.	.98.
73	768.	318.	205.	495.	45.	.62	621.	171.	110.
F.7	1120.	670.	135.	474.	21.	15.	643.	193.	125.
57	898.	4-18.	289.	450.	o	ö	. 919	. 977	146.
97	E	259.	167.	505.	52.	34.	709.	259.	167.
77	632.	182.	. 211	. 916	99	43.	698.	248.	160.
87	768.	318.	702.	621.	171.	110.	594.	-1	93.
ຂ	736.	286.	185.	632.	182.	117.	. 919	777	146.
2	70).	. 657	167.	707	152.	98.	60.5	152.	.98.
Ξ	632.	182.	117.	578.	128.	83.	•	ı	,

. June 16 - July 19: 550 ofs (355 ugd) at Chain Bridge

TABLE C.0-1 (Continued)

	:		<u>.</u>	. <u>:</u>	_:	_:		.;	<u>.</u>		_:	<u>.</u>	:	_:	<u>.</u>																	
	Bridge-	(MGD)	381.	245	=======================================	160	445	432	368	347	152	419	535	12	671	968	101	•	1	•	•	•	•	•	1	•	•	1	•	•	•	
DECEMBER	ml acc.	(c(s)	590.	379.	182.	248.	.069	670.	570.	538.	398.	650.	830.	730.	1040.	1380.	1570.	•		,	,		•	•		•	•	1	,	•	•	
	1930 obs.	(c(e)	1040.	8 29.	632.	. 869	1140.	1120.	1020.	988.	848.	100.	1280.	1180.	1490.	1830.	2020.	•	•	•	ı	•	ı	•	•	1	•	1	•	•	•	
	Br Idge	(MGD)	47.	20.	52.	103.	167.	174.	93.	93.	146.	160.	103.	160.	160.	139.	103.	146.:	215.	205.	361.	587.	277.	167.	205.	167.	185.	103.	347.	62.	=	1
NOVIEWDAR	_	(c(s)	73.	31.	80.	160.	. 558	270.	14 .	144.	. 922	248.	.091	248.	248.	215.	160.	.977	334.	318.	560.	910.	430.	259.	318.	259.	.982	160.	538.	%	17.	•
2	1930 obs.	(cfs)	523.	481.	530.	910.	709.	720.	594.	591.	676.	698.	610.	.869	.969	. 699	610.	676.	784.	768.	1010.	1360.	880.	709.	768.	709.	736.	610.	988.	546.	467.	
	r idge	(MGD)	117.	117.	98	110.	93.	47.	25.	.89	52.	43.	.29	43.	. 99	57.	52.	52.	.62	72.	62.	160.	125.	153.	167.	160.	165.	125.	25.	1 3.	72.	•
CTCHAR	-Chain B	(cfs)	182.	182.	152.	171.	144.	73.	38.	105.	60.	Ş	96.	. 99	136.	88.	8 0.	80.	45.	112.	96.	248.	193.	237.	259.	248.	. 982	193.	38.	. 99	112.	:
đ	1930 obs.	(ela)	632.	632.	602.	621.	594.	523.	488.	555.	530.	516.	5-16.	.916	586.	538.	530.	530.	495.	.795	5.16.	.869	643.	. 199	709.	698.	736.	6-13.	488.	916.	.795	•
	day	•	-	~	m	-	w	9	7	-	6	9	=	12	=	ĭ	15	91	17	18	13	20	21	77	23	1-2	25	92	17	87	ຄ	:



DISTRIBUTION OF TOTAL WASTEWATER FLOW AMONG MWA WASTEWATER TREATMENT PLANTS

Wastewater Treatment Plant 1	Fraction of Total MWA Wastewater Flow
Arlington	0.058
Blue Plains	0.759
Alexandria	0.054
Westgate ²	0.032
Piscataway	0.038
Little Hunting Creek	0.010
Dogue Creek ²	0.007
Lower Potomac	0.042
	Total = 1.000

From: GKY (1979).

THE STATE OF THE PROPERTY OF THE STATE OF TH

- 1. Listed in downstream order, with most upstream plant listed first.
- 2. These plants are no longer discharging to the estuary; conditions assumed for the modeling efforts were as shown in this table, however.

drinking water in the Metropolitan Washington Area was withheld for four days prior to discharge into the estuary through the eight MWA wastewater treatment plants. Table C.0-2 lists the capacities of these eight wastewater treatment plants as a fraction of total MWA treatment capacity based on current conditions (MPI, 1979). These fractional capacities were used in this study to determine the magnitudes of wastewater flows illustrated schematically in Figure 6.1-2. It should be noted that two of the smaller wastewater treatment plants assumed for the modeling no longer discharge to the estuary. The effect on modeling results should be negligible, however, due to the small fraction of flow involved.

WATER QUALITY COMPONENT

GOVERNING EQUATIONS AND ASSUMPTIONS

The water quality component of the DEM incorporates differential equations describing the following natural processes which affect concentrations of water quality parameters in estuarine systems (EPA, 1979):

- Advection
- Turbulent Diffusion
- Zero Order Decay
- First Order Decay
- Second Order Decay
- Biological/Chemical Transformations
- Import/Export of Mass into/out of the System

In addition to the above natural processes, the numerical solution of the advection equation introduces, an additional source of dispersion. This is one drawback of link-node models (Fischer, et al, 1979). The DEM incorporates procedures to minimize this potential source of error, however.

NUMERICAL SOLUTION TECHNIQUE

The DEM incorporates an explicit finite difference approximation of the differential equations describing the processes listed above. These equations are numerically integrated using a modified Runge-Kutta technique (EPA, 1979; Kreyszig, 1979).

INITIAL AND BOUNDARY CONDITIONS

CHARLES CARROTTE INCOMESSES INCOMESSES INCOMESSES CARROTTE

In this study, only the mass transfer of total dissolved solids (TDS) was studied. Because TDS could be treated as a conservative parameter (i.e., it undergoes no decay) the only natural processes considered in this study were advection, turbulent diffusion, and import/export of mass into or out of the system. As discussed above, the mass transport parameters of the DEM were calibrated by the EPA.

To determine the initial conditions for TDS concentrations throughout the estuary, the DEM was run for a thirty day period to establish steady state concentrations before drought water quality modeling began.

The following conditions were used to describe the boundary conditions for TDS concentrations used in this study. TDS levels in the fresh water inflow to the estuary from the Potomac River and Anacostia Rivers were assumed as listed in Table 6.1-3. The TDS levels in the seaward boundary were 30,000 mg/L. Water withdrawn from the Potomac estuary was withheld for four days prior to discharge into the estuary through the eight MWA wastewater treatment plants, as described above. The concentration of TDS was increased throughout the municipal use cycle (i.e., water treatment, municipal and industrial use, wastewater collection, and wastewater treatment) by an amount called the "use movement." The use movement for TDS, and other water quality parameters, is listed in Table 6.1-3.

REFERENCES

- Ambrose, R. B. and Roesch, S. C., "Dynamic Estuary Model Performance," J. Environmental Engr. Div-ASCE, Vol. 108, No. EE1 (Feb 1982), p. 51-71.
- Fischer, H. B., List, J. E., Koh, R. C. Y., Imberger, J., Brooks, N., Mixing in Inland and Coastal Waters, Academic Press (1979).
- GKY and Associates, "Water Quality Modeling of the Potomac River Estuary,"
 Appendix B in "Development of a Testing and Evaluation Program for the
 Experimental Estuary Water Treatment Plant, Washington, D.C.," by
 Malcolm Pirnie, Inc. for the U.S. Army Corps of Engineers, Baltimore
 District (February 1979).
- Kreyszig, E., Advanced Engineering Mathematics, Fourth Edition, John Wiley & Sons (1979).
- Roesch, S. C., Clark, L. J., Bray, M. M., "User's Manual for the Dynamic (Potomac) Estuary Model," Technical Report 63, USEPA Region III Annapolis Field Office, EPA 903/9-79-001 (January 1979).

THE PARTY CONTROL SERVICE SERVICE SERVICES OF SERVICES SE

APPENDIX D

PLANT DESIGN CRITERIA

WATER TREATMENT FACILITIES

DESCRIPTION

Plant Capacity Design Flow Rate $1900 \text{ m}^3/\text{d} (0.5 \text{ MGD})$ Normal Operating Capacity $3800 \text{ m}^3/\text{d} (1.0 \text{ MGD})$ Maximum Capacity Intake Facilities Estuary Supply Intake Screen Overall Dimensions 0.6 m by 0.9 m (2 ft by 3 ft) Bar Screen Opening 0.61 m by 0.10 m (24 in by 4 in) Fine Screen Opening 25 mm by 25 mm (1 in by 1 in) Intake Pipeline Diameter 0.25 m (10 in) Intake Pumps Type Non-clog centrifugal Number Two 22 1/s (350 gpm) Flow 20.4 m (67 ft) TDH **Motor Power** 11 kw (15 hp) Discharge Pipeline Diameter 0.2 m (8 in) Blue Plains Supply Intake Pumps Туре Non-clog centrifugal Number Four 22 l/s (350 gpm) Flow TDH 26 m (85 ft) **Motor Power** 11 kw (15 hp) Discharge Pipeline Diameter 0.2 m (8 in)

Microscreens

Number Two

Screen
Type Preassembled panels
Material Polyester cloth
Size 3.5 x 10⁻² mm (1.4 x 10⁻⁴ in)
Head Loss (maximum) 0.15 m (6 in)

DESCRIPTION

THE STATE OF THE PROPERTY WAS ASSESSED. THE PARTY OF THE

```
Drum (each)
                                                  1.6 m (5.21 ft)
    Diameter
                                                  1.3 m (4.38 ft)
     Length
                                                  6.7 \text{ m}^2 (71.7 \text{ ft}^2)
    Drum Area
                                                  2.5 \, 1/m^2 - s \, (3.7 \, gpm/ft^2)a
    Hydraulic Loading Rate
     Submergence
       Height (minimum)
                                                  74 percent
       Area
                                                  66 percent
     Speed
                                                  0.025 \text{ m/s} (5 \text{ fpm})
       Minimum
                                                  0.762 m/s (150 fpm)
       Maximum
                                                  1.1 kw (1.5 hp)
     Motor Power
  Backwash
                                                  1.7 l/s (28 gpm)
     Rate
                                                  207 kPa (30 psi)
     Presure (at nozzle)
     Number of Nozzles
                                                  Eighteen
                                                  2.2 kw (3 hp)
     Pump Motor Power
                                                  Centrifugal
     Pump Type
                                                  Ultraviolet light
  Disinfection
Blend Tank
  First Compartment
                                                  2.1 m by 2.7 m (7 ft by 9 ft)
     Dimensions
                                                  1.5 m (4.8 ft)
8.6 m<sup>3</sup> (302 ft<sup>3</sup>)
     Side Water Depth
     Volume
                                                  6.5 min
     Mean Detention Time
     Blend Mixer
                                                  Pitched blade, vertical turbine
        Type
       Impeller Diameter
                                                  1.0 m (40 in)
       Impeller Speed
                                                  28 rpm
          Low Speed
                                                  56 rpm
          High Speed
       Motor Power
          Low Speed
                                                  0.56 kw (0.75 hp)
                                                  2.2 kw (3 hp)
          High Speed
       Mixing Energy, G<sup>b</sup>
                                                  100 \, \text{sec}^{-1}
          Low Speed
                                                  400 \, \mathrm{sec}^{-1}
          High Speed
   Second Compartment
                                                  1.5 m by 2.1 m (5 ft by 7 ft)
     Dimensions
                                                  1.5 m (4.8 ft)
     Side Water Depth
                                                  4.8 \text{ m}^3 (168 \text{ ft}^3)
     Volume
                                                  3.6 min
     Detention Time
                                                  0.014 \text{ m/s} (2.8 \text{ fpm})
     Horizontal Velocity
   Total
                                                  13.3 \text{ m}^3 (470 \text{ ft}^3)
     Volume
     Detention Time
                                                  10.1 min
```

a All hydraulic loading rates and detention times given are calculated for the normal plant capacity of 1900 m³/d (0.5 MGD).

All mixing energies are approximate.

DESCRIPTION

Total

Aeration Tank 4.3 m by 4.6 m (14.25 ft by 15 ft) Dimensions 1.5 m (4.80 ft) Side Water Depth 28.9 m³ (1020 ft³) Volume Mean Detention Time 22.2 min Surface Aerator Pitched blade, vertical turbine **Type** 0.84 m (33 in) Impeller Diameter 68 rpm Impeller Speed **Motor Power** 1.5 kw (2 hp) 180 sec-1 Mixing Energy, G Rapid Mix Tanks Number Dimensions (each) 0.6 m by 0.6 m (2 ft by 2 ft) 0.93 m (3.05 ft) Side Water Depth Volume 1.39 m³ (49 ft³) Each 2.78 m³ (98 ft³) Total Mean Detention time 1.07 min Each 2.14 min Total Rapid Mixers Number Each Tank One Two Total Vertical propeller Type 0.25 m (10 in) Impeller Diameter Impeller Speed 430 rpm Mixing Energy, G $400 \, \text{sec}^{-1}$ **Flocculators** Number of Stages Two Dimensions (each) 2.0 m by 5.5 m (6.67 ft by 18 ft) 2.4 m (7.8 ft) Side Water Depth Volume 26.6 m³ (940 ft³) 51.0 m³ (1800 ft³) Each Total **Detention Time** 20.2 min Each 40.4 min Total Flocculation Mixers Number Each Tank Two

Four

DESCRIPTION

Type Pitched blade, vertical turbine Impeller Diameter 0.66 m (26 in) Impeller Speed Control Variable Range Low Speed 33 rpm **High Speed** 100 rpm **Motor Power** 1.5 kw (2 hp) Mixing Energy, G Range Low Speed 10 sec⁻¹ 200 sec⁻¹ High Speed Sedimentation Basin **Dimensions** 5.5 m by 18 m (18 ft by 59.5 ft) Average Side Water Depth 3.5 m (11.6 ft) Volume $351 \text{ m}^3 (12,400 \text{ ft}^2)$ $3.5 \times 10^5 \text{ l (92,800 gal)}$ **Detention Time** 4.5 hr 1.1×10^{-3} m/s (0.22 fpm) 19 m³/m²-d (470 gpd/ft²) Horizontal Velocity Overflow Rate Weir Length 24 m (80 ft) Weir Overflow Rate $78 \text{ m}^3/\text{m-d} (6,250 \text{ gpd/ft})$ Sludge Collector **Type** Longitudinal conveyor Sludge Pumps Number Two Туре Progressive cavity **Pump Speed** 310 rpm Flow 3.2 l/s (50 gpm) Discharge Pressure 690 kPa (100 psig) **Motor Power** 7.5 kw (10 hp) **Recarbonation Tank** First Compartment

CONTRACT TO CONTRACT TO THE STANDARY MARKET MARKETS

Dimensions 1.4 m by 1.8 m (4.5 ft by 6 ft) Side Water Depth 3.7 m (12.2 ft) 9.3 m³ (330 ft³) Volume **Detention Time** 7.2 min Second Compartment Dimensions 1.8 m by 3.2 m (6 ft by 10.5 ft) Side Water Depth 3.7 m (12.2 ft) 22 m³ (770 ft³) Volume **Detention Time** 16.6 min Total Volume $31 \text{ m}^3 (1100 \text{ ft}^3)$ **Detention Time** 23.8 min



DESCRIPTION

Predisinfection Tank Vertical Serpentine Configuration 1.4 m by 2.7 m (4.5 ft by 9 ft) Dimensions 3.7 m (12 ft) Depth $14 \text{ m}^3 (490 \text{ ft}^3)$ Volume **Detention Time** 10.6 min **Gravity Filters** Two Number 1.8 m by 3.0 m (6 ft by 10 ft) Dimensions (each) Filter Media Area $5.6 \text{ m}^2 (60 \text{ ft}^2)$ Each 11.2 m^2 (120 ft²) Total Both Filters On-Line 2.0 l/m^2 -s (2.9 gpm/ft²) Filtration Rate $1.1 \times 10^{-2} \text{ m}^3/\text{s} (174 \text{ gpm})$ Flow Rate (each) One Filter On-Line $0.24 \text{ m}^3/\text{m}^2-\text{d} (5.8 \text{ gpm/ft}^2)$ Filtration Rate 22 1/s (347 gpm) Flow Rate Media Anthracite 0.51 m (20 in) Depth 1.0 to 1.4 mm (3.9 x 10^{-2} to 5.5 x 10^{-2} in) **Effective Size** 1.5 to 1.6 Specific Gravity **Uniformity Coefficient** 1.4 Sand 0.25 m (10 in) Depth 0.4 to 0.6 mm **Effective Size** $(1.6 \times 10^{-2} \text{ to } 2.4 \times 10^{-2} \text{ in})$ 2.6 to 2.7 Specific Gravity **Uniformity Coefficient** 1.35 Gravel 0.31 m (12 in) Depth Backwash Underdrain system Precast concrete Туре $14 l/m^2 - s (20 gpm/ft^2)$ Backwash Rate (Maximum) Backwash Pumps Vertical turbine Type Two Number 76 l/s (1200 gpm) Flow TDH 8.5 m (28 ft) 11 kw (15 hp) **Motor Power** Surface Wash Agitator System Rotary straight-line Type Number (each filter) Two



DESCRIPTION

Side Water Depth

Volume

Surface Wash Pumps Vertical turbine Type Number Two Flow 3.8 1/s (60 gpm) 48 m (159 ft) TDH **Motor Power** 3.7 kw (5 hp) Filtered Water Clearwell 6 m by 10 m (20 ft by 32.5 ft) Dimensions 3.7 m (12 ft) Side Water Depth $221 \text{ m}^3 (7800 \text{ ft}^3)$ Volume Carbon Column Feed Pumps Vertical turbine **Type** Number Two 22 l/s (350 gpm) Flow 66 m (218 ft) TDH **Motor Power** 30 kw (40 hp) Downflow GAC Columns Number Three 2 m (7 ft) Diameter 5.1 m (16.75 ft) Total Height Carbon Column Bed 2.7 m (9 ft) Depth 3.6 m^2 (38.5 ft²) Area 10 m³ (350 ft³) Volume **Series Operation** 6.1 $1/m^2$ -s (9.0 gpm/ft²) Hydraulic Loading 7.5 min/column **Empty Bed Contact Time** Backwash Underdrain System Precast glazed tiles 14.1 $1/m^2$ -s (20.8 gpm/ft²) Backwash Rate (maximum) Surface Wash Agitator System Type Rotary straight-line Number (each column) One $0.75 \text{ l/m}^2\text{-s} (1.1 \text{ gpm/ft}^2)$ Surface Wash Rate Carbon Backwash Pump Vertical turbine Туре 50 l/s (800 gpm) Flow TDH 24 m (80 ft) **Motor Power** 19 kw (25 hp) Carbon Column Clearwell 4 by 7.6 m (13.2 by 25 ft) Dimensions



3.5 m (11.5 ft) 107 m³ (3790 ft³)



CERT RELEASE. MICHIGAN SERVICES MICHIGAN CONSISSION

DESCRIPTION

Disinfection Feed Pumps

Type Vertical turbine
Number Two

Flow 22 1/s (348 gpm)
TDH 8.2 m (27 ft)
Motor Power 3.7 kw (5 hp)

Chlorine Contact Tank

Type Two stage
Configuration Horizontal serpentine
Dimensions

Overall 3.7 by 6.1 m (12 by 20 ft)
Each Stage 1.7 by 6.1 m (5.6 by 20 ft)

Side Water Depth 3.5 m (11.5 ft)

Volume 3.5 in (11.5 ft)

Each Stage $36 \text{ m}^3 (1280 \text{ ft}^3)$ Total $72.5 \text{ m}^3 (2560 \text{ ft}^3)$

Total 72.5 m³ (2560 ft³)
Contact Time

Each Stage 27.7 min Total 55.3 min

Ozone Contact Tank

Type Two stage
Configuration Vertical serpentine

No. Passes Per Stage Four Dimensions

 Overall
 2.4 by 3.7 m (8 by 12 ft)

 Each Stage
 1.2 by 3.7 m (4 by 12 ft)

Side Water Depth 3.7 m (12 ft)

Each Stage 16.4 m³ (580 ft³)
Total 33 m³ (1160 ft³)

Contact Time
Each Stage 12.4 min

Total 24.8 min

Finished Water Clearwell

Dimensions 2.4 by 6.1 m (12 by 20 ft)

Side Water Depth 3.5 m (11.5 ft)
Volume 78 m³ (2760 ft³)



CHEMICAL HANDLING SYSTEM FACILITIES

DESCRIPTION

Commercial Alum System (Liquid)

Commercial Alum Tank

Diameter

Height

Capacity

Commercial Alum Pumps

Type

Number

Pump Speed, max

Pump Speed, max

Liquid)

2.3 m (7.6 ft)

5.5 m (18 ft)

22.4 m³ (790 ft³)

Positive displacement, diaphragm

Two

116 strokes/m

Feed Rate, max

0.027 1/s (25.6 gph)

Discharge Pressure, max

Feed Range Adjustment Ratio

Motor Power

0.027 1/s (25.6 gph)

690 kPa (100 psig)

25:1

0.19 kw (0.25 hp)

Commercial Lime System

Commercial Lime Storage Bin

Diameter 2.7 m (9 ft)

Total Volume 41 m³ (1440 ft³)

Rotary Airlock Feeder

Diameter 0.15 m (6 in)

Speed 2.5 rpm

Volume 6.5 x 10⁻³ m³/rev (0.23 ft³/rev)

Capacity at 75% Eff. Rating 2.04 x 10⁻⁴ m³/s (26 ft³/hr)

Motor Power

Commercial Lime Blower

Specified Capacity
Capacity
Discharge Pressure
Blower Speed

0.18 kg/s (1440 lb/hr)
0.15 m³/s (325 cfm)
17.3 kPa (2.5 psig)
4500 rpm

Blower Speed 4500 rpm
Motor Power 5.6 kw (7.5 hp)
Lime Day Hopper

 Diameter
 1.2 m (4 ft)

 Total Volume
 2.3 m³ (82.3 ft³)

Rotary Inlet Weighbelt Feeder

Rotary Inlet

Diameter 0.15 m (6 in)

Speed Variable

Capacity 7.8 kg/min (1000 lb/hr)

Speed Range Adjustment Ratio 10:1

Motor Power 0.19 kw (0.25 hp)
Gravimetric Feeder

 Width
 0.31 m (12 in)

 Belt Speed (constant)
 9.1 x 10⁻³ m/s (1.8 fpm)

 Capacity
 7.8 kg/min (1000 lb/hr)

Motor Power 0.37 kw (0.5 hp)

DESCRIPTION

Pressure Range

Capacity

TREEZERER SEPRESSO SERVICES (NEWSFRE) RESERVED

Lime Slaker **Type** 7.8 kg/min (1000 lb/hr) Capacity 2/1 Water/Quicklime (CaO), by weight Slurry Concentration (CaO), by weight 33% 0.37 kw (0.5 hp) **Motor Power** Lime Slurry Tank 1.2 m (4 ft) Diameter $2.5 \text{ m}^3 (88 \text{ ft}^3)$ Volume Mixer Vertical pitched blade **Type** 1.5 kw (2 hp) **Motor Power** Lime Slurry Pumps Positive displacement, diaphragm **Type** Number Two Number of Heads 88 strokes/min Pump Speed, max 1.06 l/s (1010 gph) Feed Rate, max 483 kPa (70 psig) Discharge Pressure, max Feed Range Adjustment Ratio 25:1 1.5 kw (2.0 hp) **Motor Power** Polymer Feed System Polymer Feed Equipment Mix Tank 0.76 m (2.5 ft)Diameter $0.35 \text{ m}^3 (12.4 \text{ ft}^3)$ Capacity Mixer Propeller Type Day Tank 0.76 m (2.5 ft) Diameter 24.5 1 (93 gal) Capacity Polymer Pump Positive displacement, diaphragm Type Four Number 116 strokes/min Pump Speed, max 0.027 l/s (25.6 gph) Feed Rate, max 1587 kPa (230 psig) Discharge Pressure, max Feed Range Adjustment Ratio 25:1 **Motor Power** 0.19 kw (0.25 hp)Commercial Carbon Dioxide System Liquid Carbon Dioxide Tank 2.0 m (6.5 ft) Diameter $30 \text{ m}^3 (1048 \text{ ft}^3)$ Volume

1725 to 2760 kPa (250 to 400 psig)

14515 kg (32 ton)

DESCRIPTION

Chlorination System

Chlorinators

Number Two

Capacity, max 36 kg/day (80 lb/day)

Chlorine Storage

Type Cylinder Capacity 68 kg (150 lb)

Ozone Generator (prior to modification)

Type Horizontal Tube, Corona Discharge

Number One

Capacity, max 37.6 kg/day (83 lb/day) of ozone

Air Flow Rate, max

Pre Disinfection 0.016 standard m³/s (33.5 scfm)
Post Disinfection 0.047 standard m³/s (100 scfm)

SOLIDS HANDLING AND CHEMICAL RECOVERY FACILITIES

Backwash Handling System

Backwash Holding Tank
Dimensions 7.6 by 7.6 m (25 by 25 ft)

 Side Water Depth
 3.7 m (12 ft)

 Volume
 205 m³ (7225 ft³)

Rake Mechanism

 Diameter
 7.6 m (25 ft)

 Speed
 0.01 rpm

Motor Power 0.25 kw (0.33 hp)

Discharge Pump

Type Submersible non-clog centrifugal

Number One

 Flow
 8.2 1/s (130 gpm)

 Discharge Pressure
 9 m (30 ft)

 Motor Power
 1.1 kw (1.5 hp)

Sludge Storage/Carbonation

Diameter 3.0 m (10 ft)
Side Water Depth 4.0 m (13 ft)
Volume 29 m³ (1021 ft³)

Volume Mixer

Type Flat blade turbine

Number One

Impeller Diameter 1.14 m (3.75 ft)

Impeller Speed 84 rpm

Motor Power 7.5 kw (10 hp)
Mixing Energy, G 400 sec⁻¹

Thickened Sludge Pumps

Type Progressive cavity

Number Two

Pump Speed 0.87 to 6.1 rps (52 to 364 rpm)
Flow 0.38 to 3.8 l/s (6 to 60 gpm)

Discharge Pressure 690 kPa (100 psig)
Motor Power 7.5 kw (10 hp)

APPENDIX E

PLANT OPERATIONS DATA

SECTION 1

AVERAGE CHEMICAL USE AND DAILY AVERAGE FLOW DATA FOR OPERATIONAL PHASES IA, IB AND II

TABLE E.1-1

ANN THE PERSONAL PROPERTY OF THE PERSON STATES OF THE PERSON STATES OF THE PERSON STATES AND THE STATES.

CHEMICAL USE DATA - PHASES IA AND IBI

Arithmetic Mean BE/L IB IB IA Value Value Value Value Value Value Value Value Value 1B 50.7 53.9 19.4 45.1 NU ² 0.123 0.09 - 0.244 0.229 0.02 0.09 - 0.244 0.229 0.02 0.09 - 0.40 - 4.26 4.26 4.26 4.26 4.26 4.26 3.75 3.77	1A+ 1B 1B 0.123 0.123 0.184 0.229 17.4 1.70 2.27 4.26 4.51	um Maximum											
118 128 138 138 138 138 138 138 138 138 138 13	Arithmetic Me 1A IB IB 54.8 50.7 0.123 NU ² 0.184 NU 0.198 0.244 15.1 20.9 nate 1.70 NU e 2.27 NU NU 4.26 11.07 4.26 2.87 3.75												
		Arithmetic Mean mg/L	11	50.7	•	25	777	20.02	N E	Ž	4.24	7.7	3.75

See Table 7.1-1 for specific operating period.
 NU = Chemical Not Used during this operational phase.

TABLE E.1-2
CHEMICAL USE DATA - PHASE II

Parameter	Average mg/L	Minimum mg/L_	Maximum mg/L
Lime (as CaO)	77.7	34.2	155.0
FeCL ₃	2.2	1.5	4.3
Ozone	1.3	0.24	2.7
NH ₃ (as N)	1.6	0.30	3.4
CO2	230.4	76.2	450.0
Final Chlorine	4.9		-

^{1.} See Table 8.1-1 for special operating periods.

TABLE E.1-3

DAILY AVERAGE FLOW DATA - PHASES IA AND IB (gpd)

PARAMETER		Average		Minimum	anne	Mexi	
	4	8	IA & IB	41		4	8
Blended Influent Est/Forebay, %	522,916	505,647	517,714	180,900	00	597,599	536,100
Filter Backwash	11,450	11,434	11,450	2,155	9,160	19,200	11.940
Carbon Column Backwash!	10,942	16,920	110,11	2,600	5,640	16,130	23,900
Finished Water	501,965	508,758	503,483	175,799	473,600	569,250	545,900

1. Average for each backwash.

TABLE E.1-4

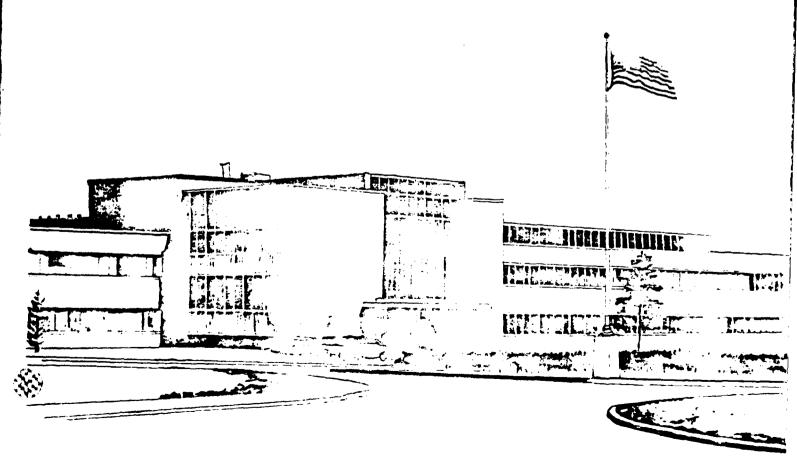
DAILY AVERAGE FLOW DATA - PHASE II (gpd)

PARAMETER	Average	Minimum	Maximum
Blended Influent	503,997	0	550,400
Est/Forebay, %	50	0	100
Filter Backwash	13,609	8,942	20,114
Carbon Column Backwash	11,880	6,829	17,160
Finished Water	230,466	0	272,160

SECTION 2

SUMMARY REPORT OF REACTIVATION OF EEWTP SPENT GRANULAR ACTIVATED CARBON BY ICI AMERICAS INC., SEPTEMBER, 1981 AND APRIL, 1982





REGENERATION OF HYDRODARCO 816 FROM BLUE PLAINS PILOT PLANT, D.C.

(JAMES MONTGOMERY, CONSULTING ENGINEERS, INC.)

Submitted: A. P. Ferrara September 1981

REGENERATION OF HYDRODARCO 816 FROM BLUE PLAINS PILOT PLANT, D.C.

A. Objective:

To provide custom reactivation at DXL of spent Hydrodarco 816 taken from 2½ columns at EEWTP at Blue Plains, D.C. for James Montgomery Engineers.

B. Summary:

A shipment of 31,820 pounds, as is of spent Hydrodarco 816 was received at DXL on June 19, 1981 from Blue Plains, D.C. The carbon was regenerated at the DXL Pilot Plant June 22 to June 25 in the Single Tube Experimental Unit. The regenerated carbon (14,080 pounds d.b.) was returned to Blue Plains by J. Miller Express. A regeneration yield of 93.2% (correcting for 5% VCM difference in spent and regenerated carbon) was achieved.

Standard analytical testing of spent, regenerated and virgin material was made at DXL and provided in this report.

The regeneration was witnessed by Mr. Tom Sadick of James Montgomery Engineers.

C. Results and Discussion:

Table 1 provides material balance data collected on the regeneration run. Volumetric yield was calculated at 91.1% and 93.2% corrected for 5% VCM in spent material fed to the furnace.

Laboratory analysis of the virgin material supplied from Blue Plains, spent, and regenerated product are presented in Table 2. Table 3 gives typical properties of virgin Hydrodarco 816 and average of Hydrodarco 816 shipped from Marshall Plant to Blue Plains. Property determinations on composite shipment to Blue Plains run about February 1980 included moisture. pH. VFD, Iodine Number and Abrasion Number.

The virgin carbon sample from Blue Plains pilot plant sampled from an adsorber as a Control may have been partially spent in the column before sampling. This likely occurred within the long period between the time installed (February 1980) and when the carbon was sampled (about May 1981). According to Tom Sadick, some of the original column linings had deteriorated and had to be replaced. There is a possibility that some adsorption of some organics leached out may have been adsorbed by the GAC while standing for 15 months before start-up. This is shown in Table 2 by the high V.C.M. (11.1%) and V.F. Density (0.45~gms/ml) and relatively low Molasses RE (90) and phenol number In addition small pieces of gravel and sand-like particles were observed in the carbon which likely contributed to the originally high VF density determined in the Blue Plains virgin sample.

Table 3 provides properties for typical production of Hydrodarco 816 along with various property measurements representing an average of three shipments to Blue Plains.

Figure 1 and 2 of the Appendix show surface area and pore size distribution comparisons for spent, regenerated, and virgin (Blue Plains) samples. For additional information a typical pore size distribution curve for Granular Darco has been provided and represented as a dotted line in Figure 2.

As seen in Figure 2 the pore volume of the virgin carbon from Blue Plains was substantially less than its regenerated counterpart at pore sizes >500°A and more comparable to the spent material. This may in part reflect the adsorption of leached organics from the lining as described earlier. The pore size curve for the regenerated product shows good restoration of pore volume and is comparable to typical virgin carbon (dotted line). All three products appear more comparable in surface area distribution (Figure 1).

ARROW DESIGNATION OF STREET, S

Average operating conditions in the STEU furnace are presented in Table 4. As seen in this table a gradual decrease in lower hearths temperature and increase in feed r.p.m. was made in an effort to lower the Iodine Number. However, V.F. Density, the primary regeneration control parameter in furnace product, did not fall off as normally expected to levels typical of virgin carbon (0.40-0.42). These contrasting data are likely attributed to not having a good reference sample of virgin carbon as a Control and resulted in a slight over-reactivation of the product.

This was reflected in the somewhat higher Iodine Number of the composite regenerated product (695) vs. material shipped to Blue Plains (617 avg.) as seen in Tables 2 and 3.

The Molasses RE (104), Tannin Value (225), VCM (5.1), Phenol Value (2.9), and Total Pore Volume (0.9) of the regenerated product overall were comparable to that expected for typical virgin carbon (Tables 2 and 3).

MONORAL SECURITY MANAGEM LANGEST MANAGEM MANAG



REGENERATION YIELD DATA

Spent Hydrodarco 816 received as is at 4/.0% Moisture	31,820 lbs.
Spent Hydrodarco 816 received d.b.	16,865 lbs.
Loss (truck wash out + transfer from storage pad to	•
feed levels), d.b.	724 lbs.
Feed to furnace 29,347 lbs., as is, at 45.0%	
moisture, d.b.	16,141 lbs.
Less charge head seal leakage, d.b.	180 lbs.
Net feed to furnace, d.b.	15,961 lbs.
Net feed to furnace, volume	564 ft. ³
Packed out Regenerated Hydrodarco 816, d.b.	14,080 lbs.
Samples and packaging spillage	52 lbs.
Net regenerated Hydrodarco 816, d.b.	14,132 lbs.
Net regenerated Hydrodarco 816, volume	514 ft. ³
Yield, % by volume	91.1%
Yield, % by weight corrected for 5% VCM loss,	
$14,132 \div (15,961 \times 0.95) \times 100 =$	93.2%
Average feed rate to furnace, as is	506 lbs.
Average discharge rate from furnace, d.b.	244 lbs./hr.
	•

TABLE 2

PROPERTY DATA ON HYDRODARCO 816

(virgin from Blue Plains, spent, regenerated)

Carbon	Virgin (from Blue Plains)	Spent	Regen.*
Moisture, as received from Blue Plains, % Moisture, % V.F.A. Density, d.b., gm/ml Iodine Number Molasses RE VCM, d.b., % Ash, d.b., % Abrasion Number (% Retention - Min., stirring Abrasion Test) Total Pore Volume, ml/gm Phenol Value (modified), gms/l. Tannin Value, ppm Avg. Particle Diameter, m.m. Effective Size, m.m.	>60.0 1.4 0.452 530 90 11.1 13.6 80.2 0.81 3.4 202 1.50 0.95	486 94 10.0 12.1 77.0	
Uniformity Coeff.	1.7	1.8	1.9

^{*}Based on regenerated carbon composite

TABLE 3

PROPERTY DATA OF TYPICAL VIRGIN HYDRODARCO 816 AND AVERAGE OF 3 SHIPMENTS TO BLUE PLAINS

	Typical Virgin Hydrodarco 816	Hydrodarco 816, Avg. of Three Shipments to Blue Plains
	5-6	5.9
Moisture (%)	4.5-7.5	5.3
pH (water extract)		
V.F. Density gms/ml, d.b.	0.400	0.395
Abrasion No. (% Retention -		
Min., stirring abrasion		
tests)	80	81
Iodine Number	600	617
	100	•
Molasses RE	5-7	_
VCM, d.b., %		
Ash, %, d.b.	12-14	
Tannin Value, ppm	250	-
Phenol Value (modified),		
g/1	3.0	-
Total Pore Volume, ml/gm	0.9-1.0	-

Table 4.

(Steam Flow = 100 lb./hr.; Tube Slope = 0.150 inches/ft.)

	6-22-81 6 pm-12 M	6-23-81 12 M- 1 pm	6-23-81 1 pn- 12 M	6-24-81 12 M- 8 am	6-24-01 8 am-12 M	6-25-81 12 M-5 am
Furnace Temperature. Oc.			· .			
TC-1	S	Н	9	σ	Ø	σ
10-2					_	0
	1030	066	916	940	940	940
Bed Temperature, C.						
	$\boldsymbol{\varsigma}$	\mathbf{c}	~	m	4,	4
TC-2	9	S	4	9	9	S
TC-3	σ	9	4	~	~	~
TC-4	9	2	2	Н	0	$\boldsymbol{\vdash}$
TC-5	S	2	ぜ	N	$\boldsymbol{\vdash}$	œ
TC-6	n	S	S	œ	œ	~
Tube, rev./min.	4.0	4.2	4.5	4.5	5.0	5.0
Feed, rev./min.	•	•	•	•	•	•
Grab Samples		` . .			•	
V.F.A. Density, g./ml.	1	9.446	0.428	0.442	1	1
Iodine Number	1	0	9	σ	i i	i 1
				,		

APPENDIX

STATE COURTY SOURCE WASHING SCHOOL BERNER 1750

TABLE 4-1

·	المستعدد والمستعدد والمستعد والمستعدد والمستعد	LAB NO. 22.J	UN 31-4
····	. RADII RANGE ANGSTREMS	Hs INTRUCION.	
	•		PORE VOLUME ML./G.
· .	50,000 - 39,800	0.0213	
		A A1A0	
	25,100 - 15.800	0.0038	· management of the same of
	15,800 - 10,000	0.0101	
	10,000 - 6,300	0.0602	
	6,300 - 3,980	0.0257	
···	3,980 - 2,510	0.0677	
	2,510 - 1,580	0.0272	
	1,580 - 1,000	0.0082	
	1,000 - 630	0.0065	
:	630 - 398	0.0091	
•	398 - 251	0.0106	- 0.0069
	251 - 158	0.0129	0.0140
	158 - 100	0.0420	0.0139
	100 - 63	0.0360	0.0394
***************************************	63 - 39.8	0.0854	0.0617
	39.8 - 35	0.0107	- -
<u> </u>	39.8 _ 25.1	· · · · · · · · · · · · · · · · · · ·	0.0869
	25.1 - 15.8		0.1616
	15.8 - 10.0		0.0947

TABLE, B-1

CUMULATIVE FORE VOLUME IN ALL FORES HAVING RADII LESS THAN THE INDICATED SIZE *

PORE RADIUS	TAR " NOWRFK
ANGSTROMS	22 JUN 81-4
50,000	0.8130
39,800	0.7917
25,100	
15,800	0.777.1
10,000	0.7670
6,300	0,7068
3,980 •	0.6811
2,510	0.6134
1,580	0.5862
1,000	0.5780
630	0.5715
398	0.5624
251	0.5518
158	0.5389
	0.4969
63	0.4575
39.8	0.3958
25.1	0.3089
15.8	0.1473
10	0.0526
•	•

DATA LESS THAN 100 ANGSTROMS FROM NITROGEN DESORPTION

TABLE C-1

PORE VOLUME DISTRIBUTION DATA * LAB NO. 22 JUN 81-4

: PORE RADII IN A	NGSTROMS	FORE VOLUME, ML./G. DRY BASIS		
INCREHENTAL	CUMULATIVE	INCREMENTAL	INCREMENTAL	ÇUMULF
_50,000 <u>-39,800</u>	39,800	. 0213 و 0	0.009	2,0
39.800 - 25.100	25,100		0.007	0.0
25 • 100 - 15 • 800	15,800	0.0038	0.004	0.0
15-800 - 10-000			0.016	0.0
10,000 - 6,300	6,300	0.0602	0.148	0.1
6,300 - 3,980 <u>-</u>	3,980	0.0257	0.100	0.2
3,980 - 2,510	2,510	0.0677	0.417	0.7
2,510 - 1,580	1,580	0.0272	0.266	0.5
1.580 - 1.000	1,000	0.0082	0.127	0 بخت
1,000 - 630	630	0.0065	0.160	
630 - 398	. 398	0.0091	0.354	1.6
398 - 251	251	0.0106	0.653	2.2
<u> 251 - 158</u>	158	0.0129	1.262	3.5
158 - 100	100	0.0420	6.512	10.0
100 - 63	63	0.0394	9.62	19.6
63 - 39.8	39.8	0.0617	23.88	43.5
39.8 - 25.1	25.1	0.0569	53.31	96.8
25.1 - 15.8 .	15.8	0.1616	157.20	254.0
15.8 - 10.0	10.0 .	0.0947	146.11	400.1
RESIDUAL	SURFACE AREA			278
* DATA LESS THAN 1	LOO ANGSTROMS	FROM NITROGEN DESOR	FTION. ·	
• • • • • • • • • • • • • • • • • • • •	· · · · · · · · · · · · · · · · · · ·	entre de la companya br>Canada de la companya de la company	i Annon an annon communication and an annon an an Annon an annon an an	
And the second plan recognition of the second secon				

REGENERATED HD-816

TABLE A-2

·			LAR NO. 25 J	UN 81-1	· ••
		KANGE			
· · · · · · · · · · · · · · · · · · ·	ANGSTR		INTRUSION	DESORPTION	
	·	7 au 1 mar 1 c .		FORE VOLUME ML,/G.	
	5 2 222	70 500		and the no sub-the net per par par our no no	. .
• •	50,000 - 39,800 -	25,100	0.0264	· · · · · · · · · · · · · · · · · · ·	
•	25,100 -		0.0016		
			0.0033		
		10,000	0.0128 '		
•	10,000 -	6,300	0.0062	•	
	6,300 -		8080·O		٠.
	3,980 -	2,510	0.0390	· · · · · · · · · · · · · · · · · · ·	
	2,510 -		0.0367		
	1,580 -		0,0241		
• .	1,000 -	630	0.0186	•	
•	. 630 -	398	0.0491		
	398		0.0179	0.0079	
	251 -		L 0.0222	0.015	
المنا المستعدد الأستسان أأسا	, 58 -		0.0339	0.0156	
ta yan daya adadaan ahara waxaa ii waxaa a			,0.0577	0.0299	
•	63 -	39.8	0.0722	0.0717	
	. 39.8 -	35 ·	0.0304		
en e	39 • 8	25.1		0.1111	
	25.1 -	15.8		0.1767	
	15.8 -		The second secon		

TABLE B-2

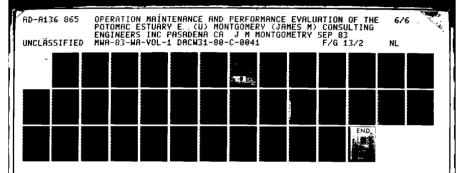
	EFFECERCESES	
ANGSTROMS		
- nnosinons		
50,000	0.8780	•
39,800	. 0.8716	
25,100	0.8700	
15,800	0.8667	
10,000	0.8539	
6,300	0.8477	or many recommendation of the first contraction of the first contractio
3,980	0.7669	
2,510	0.7279	•
1,580	0.6912	
1,000	0.6671	
630	0.6485	
398	0.5994	
25%	0.5815	
ં હ	0 · 5593,	•
100	0.5254	
. 63	0.4955	والمراجع والمستوان
39.8	0.4238	and the second s
25.1	0.3127	
15.8	.0.1360	
10	0.0439	•

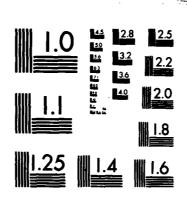
* DATA LESS THAN 100 ANGSTROMS FROM NITROGEN DESCRITION

TABLE C-2

PORE VOLUME DISTRIBUTION DATA * LAB NO. 25 JUN 81-1

PORE RADII IN A	ANGSTROMS	PORE VOLUME, ML./G DRY BASIS	DRY, 3	
INCREMENTAL	CUMULATIVE	INCREMENTAL	INCREMENTAL	. .
50.00039.800	39,600			
39,800 - 25,100	25,100	0.0013 ,	. 0.001	C
5,100 - 15,800	15,600	0.0033	0.003	. 0
5,800 - 10,000 -	10,000	0.0128	0.020.	C
0,000 - 6,300	&,300	0.0362	0.015	C
6,300 - 3,980	3,980	0.0808	0.314	Ö
3,980 - 2,510	2,510	0.0390	0.240	0
2,510 - 1,580	1,580	0.0367	0.359	(
1,580 - 1,000	1.000	0.0241	0.374	
1,000 - 630	630	0.0156	0.456	
630 - 398	378	0.0491	1.911	•
398 - 251	251	0.0179	1.103	•
251 - 158	158	0.0222	2.171	(
158 - 100	100	0.0339	5.256	1
100 - 43	· 3	0.0279	7.30	1
63 - 39.8	39.8	0.0717	27.74	. 4
39.6 - 25.1	25.1	0.1111.	68.11	11
25.1 - 15.8	15.8	0.1767	171.90	. 28
15.8 - 10.0	10.0	0.0921	142.02	42
RESIDUAL	SURFACE AREA			26





#1.

TOTAL STREET STREET, S

MICROCOPY RESOLUTION TEST CHART

NATIONAL BUREAU OF STANDARDS-1963-A

SPENT HD-816

TABLE A-3

F	ORE VOLUME BY MERCURY I	NTRUSION AND NI	TROGEN DESCRPTION	•
=		LAB NO. 25. JL	IN 81-2	. <u></u> .
	RADII RANGE ANGSTROMS	Ha INTRUSION .	N2 DESORFTION	•
-	/ !	INCREMENTAL D.B. N	FURE VOLUME	
•	50,000 - 39,800	0.0237	and the first supplies to the	
•	39,800 - 25,100			
	25,100 - 15,800	0.0135	and the second s	
	15,800 - 10,000	0.0110		•
	10,000 - 6,300	0.0079 .	•	
•	6,300 - 3,980	0.0040		·
	3,980 - 2,510	0.0206		
	2,510 - 1,580	0.0678		
	1,580 - 1,000	0.0143	·	
•	1,000 - 630			
•	630 - 398	0.0218	•	
	398 - 251	0.0068	0.0033	
	251 - 158	0.0294	0,0156	
	158 - 100	0.0214	_0.0164	
	100 - 63	0.0245	0.0335	
•	63 - 39.8	0.0738	0.0561	
	39.8 - 35	0.0153	-	•
	39.8 - 25.1		0.1248	
-	25.1 - 15.8		. 0.1857	
	15.8 - 10.0			•

TABLE 3-3

CUMULATIVE FORE VOLUME IN ALL MORES HAVING RADII LESS THAN THE INDICATED SIZE *

PORE RADIUS	LAB NUMBER
ANGSTROMS	25 JUN 81-2
50,000	0.7840
.39,800	0.7603
. 25,100	0.7528
15,800	, 0.7393
10,000	0.7283
6,300	0.7204
3,980	0.7164
2,510	0.6958
1,580	0,6280
	0.6137\
630 398	0.5943
398	0.5725
251	0.5657
158	0.5363
100	0.5149
63	0.4814
39.8	<u>, </u>
25.1	0.3005
15.8	0.1148
10	0.0601
. 💉	

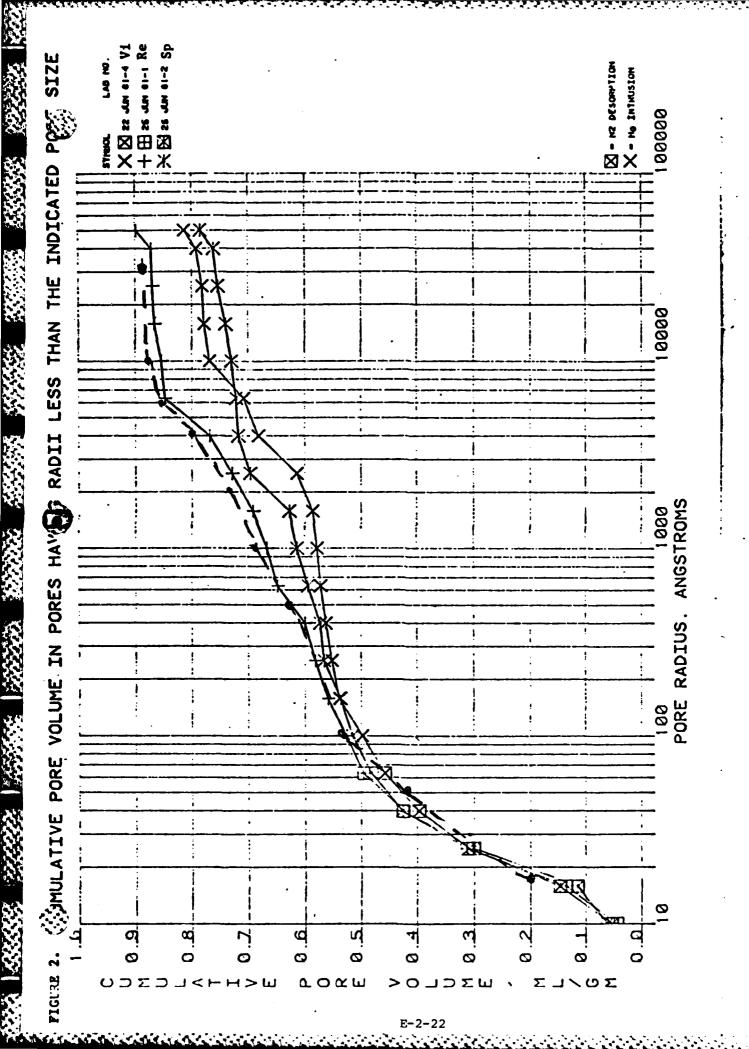
* DAJA LESS THAN 100 ANGSTROMS FROM NITROGEN DESORPTION

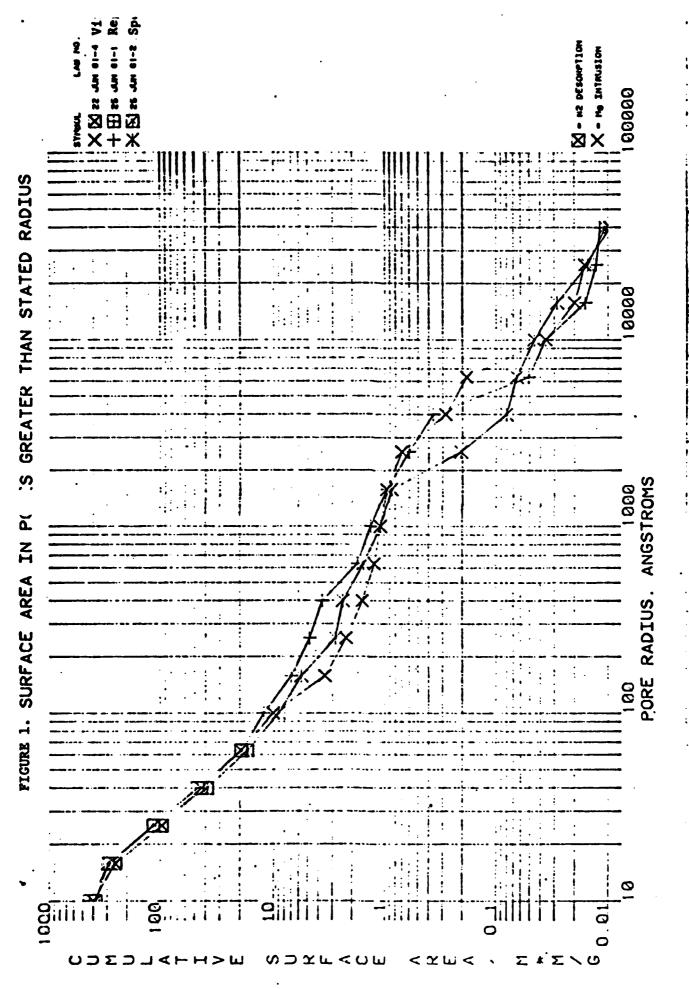
TABLE C-3

PORE VOLUME DISTRIBUTION DATA *LAB NO. 25 JUN 81-2

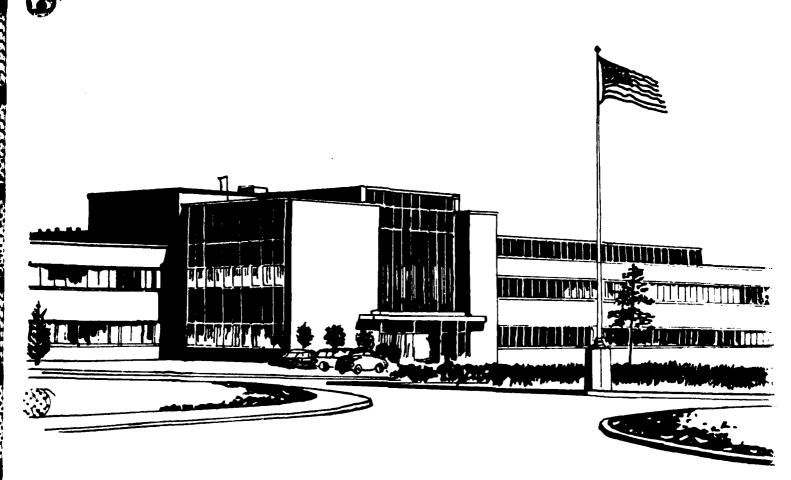
PORE RADII IN ANGSTROMS		PORE VOLUME, ML./G. DRY BASIS		CALC. SURFACE AREA, NA DRY BASIS	
INCREMENTAL	CUMULATIVE	INCREMENTAL .	INCREMENTAL	CUMUL	
50,000 - 39,800	39,800	0.0237	0.011	0.(
39,800 — 25,100 _.	25,100	0.0075	0.005	0.0	
25,100 - 15,800	15,800	0.0135	0.013	0.0	
15,800 - 10,000 <u> </u>	10,000	0.0110	0.017	0.0	
10,000 - 6,300	6,300	0.0079	0.019	0.0	
6,300 - 3,980	3,980	. 0.0040	. 0.016	0.0	
3,980 - 2,510	2,510	0.0206	0.127	0.2	
2,510 - 1,580	1,580	0.0678	0.663	0.5	
1,580 - 1,000	1,000	0.0143	0.222	1	
1,000 - 630	630	0.0194	0.476	1	
630 - 398	398	0.0218	0.848	2.4	
398 - 251	251	8800.0	0.419	2.8	
251 <u></u> 158	158	0.0294	2.875	5.7	
158 - 100	100,	0.0214	3.318	9.0	
100 - 63	. 6 3	0.0335	8.18	17.3	
63 - 39.8	39.8	0.0561	21.71	38.9	
39.8 - 25.1	25.1	0.1248	76.52	115.4	
25.1 - 15.8	15.8	0.1857	180.69	296.1	
15.8 - 10.0	- 10.0	0.0547	84.35	380.4	
RESIDUAL	SURFACE AREA			196	

·









SECOND REGENERATION OF HYDRODARCO 816
FROM BLUE PLAINS PILOT PLANT, D.C.
(James Montgomery, Consulting Engineers, Inc.)

A. Peter Ferrara Product Development Department

April 29, 1982

SAME SAME DESCRIPTION SAME PROPERTY

SECOND REGENERATION OF HYDRODARCO 816 FROM BLUE PLAINS PILOT PLANT, D.C.

A. Objective:

To provide the second custom reactivation at DXL of spent Hydrodarco 816 from carbon columns at the EEWTP at Blue Plains, D.C. for James Montgomery Engineers.

B. Summary:

MOREOWN WARRENT TO THE WAY OF THE CARREST OF

CANNOTA CHEMICAL MODERN MENDER SERVICE SOCKERS AND

A shipment of 42,880 pounds, as is, of spent Hydrodarco 816 was received at DXL on December 21, 1981 from the Blue Plains EEWTP. Carbon was regenerated at the DXL Pilot Plant from January 4 to January 7 in the Single Tube Experimental Unit. The regenerated carbon (20,040 pounds d.b.) was returned to Blue Plains by J. Miller Express.

Standard analytical testing of spent, regenerated and virgin material was made at DXL and provided in this report.

Overall regeneration results appeared favorable with high furnace yield (98.5% correcting for VCM difference in spent and regenerated carbon), and good restoration of carbon properties.

C. Results and Discussion:

Material balance data are provided in Table 1. Volumetric yield was calculated at 96.5% and 98.5% corrected for VCM differences of spent and regenerated products.

Analyses of virgin material from Blue Plains, spent, and regenerated products are given in Table 2. Table 3 gives typical properties of virgin Hydrodarco 816 and average of Hydrodarco 816 shipped from Marshall Plant to Blue Plains.

The gravel and sand-like particles which were observed in virgin carbon for the first regeneration were not observed in the latest virgin carbon sample from Blue Plains. However, this material still had a relatively high V.C.M. (9.3%) and V.F. density (0.430 gms per ml) compared to typical virgin Hydrodarco 816 (5-7% V.C.M. and 0.400 V.F.D. - Table 3). Interestingly, the Molasses RE (118) and Tannin Value (191) of the virgin sample from Blue Plains was unusually better than would have been expected for typical virgin product. This may have been related to sampling methods. The total pore volume (0.92 ml/gm) was similar to virgin material.

As with the first regeneration, V.F. density, a primary regeneration control did not fall off to levels typical of virgin carbon (0.40-0.42).

Regenerated product yield (98%) was excellent and over-reactivation did not appear to occur during this second run.

As seen in Table 2 the regenerated product overall showed favorable restoration of product properties. Iodine Number (619), Phenol Value (3.2), V.C.M. (4.3), and Tannin (200) appeared representative of typical virgin carbon.

haddada teedaya 1888888 hadada dadda badada 8860ee qasaga agaga gasaga

Overall shown in Figure 1 the pore volume distribution of the regenerated product is comparable to typical virgin carbon with slightly less volume in pores >3000°Ar which could account for the marginally lower Molasses RE (92) relative to virgin carbon. The dotted lines represent the pore volume distribution from the first regenerated carbon and a typical virgin Hydrodarco 816. The somewhat larger pore volume of the latest virgin sample from Blue Plains in the range >1000 compared to typical virgin material possibly could be explained by the higher Molasses RE (118) of this material.

The spent carbon feed to the furnace appeared to be somewhat more spent than the first regenerated feed. This was observed by the lower Iodine Number (401) and Molasses RE (81) relative to the previous spent feed (486 and 94 respectively). Pore volume distributions are still being run on this material and data will be provided upon receipt from DXL.

TABLE 1

REGENERATION YIELD DATA

Spent Hydrodarco 816 received as is at 45.4% moist.	42,880 lbs.
Spent Hydrodarco 816 received d.b.	22,555 lbs.
Loss (truck wash out and transfer from storage pad to	
feed levels) d.b.	619 lbs.
Feed to furnace (as is)	40,176 lbs.
Net feed to furnace d.b.	21,936 lbs.
Net feed to furnace, volume	736 ft ³
Packed out Regenerated Hydrodarco 816, d.b.	20,040 lbs.
Samples and packaging spillage	48 lbs.
Net regenerated Hydrodarco 816, d.b.	20,088 lbs.
Net regenerated Hydrodarco 816, volume	710 ft ³
Yield, % by volume	96.5%
Yield, % by weight corrected for 6.7% VCM diff.	98.5%

TABLE 2

PROPERTY DATA ON HYDRODARCO 816 (virgin from Blue Plains, spent, regenerated)

Carbon	Virgin (from B.P.)	Spent	Regen.*	
Moisture (oven), %	14.0	45.4		
V.F.A. Density, d.b., gm/ml	0.430	0.478	0.453	-
Iodine Number	538	401	619	Ę
Molasses RE	118	81	92	
VCM, d.b., %	9.3	11.0	4.3	
Ash, d.b., %	14.4	12.8	13.2	
Abrasion Number (% Retention - Min.,				
stirring Abrasion Test)	79.1	78.5	80.2	
Total Pore Volume, ml/gm	0.92	0.75	0.83	
Phenol Value (modified), gms/1	3.5	-	3.2	
Tannin Value, ppm	191	-	200	
Avg. Particle Diameter, m.m.	1.50	1.45	1.38	

TABLE 3

PROPERTY DATA OF TYPICAL VIRGIN HYDRODARCO 816
AND AVERAGE OF 3 SHIPMENTS TO BLUE PLAINS

Ø

	Typical Virgin Hydrodarco 816	Hydrodarco 816, Avg. of Three Shipments to Blue Plains
Moisture (%)	5-6	5.9
pH (water extract)	4.5-7.5	5.3
V.F. Density gms/ml, d.b.	0.400	0.395
Abrasion No. (% Retention -		
Min., stirring abrasion		
tests)	80	81
Iodine Number	600	617
Molasses RE	100	-
VCM, d.b., %	5-7	•
Ash, %, d.b.	12-14	-
Tannin Value, ppm	250	-
Phenol Value (modified),		
g/1	3.0	-
Total Pore Volume, ml/gm	0.9-1.0	-

			1		į	!	
•							
:	İ	کے کہ	226	1:11		0 0	4.52
	:	7-8	800	250	202	00	7.7
<u>!</u>	7025	1-1	444	222	700	0 0	4.4
	CONPITIONS	£ ,	722	222	700	0 0	2,2%
ij	•	1-6-9 r	278	37.10		0 0	457
	OPERATIVE	1-1	264	2000	750	00	473
SLE	2000	=======================================	272	250	220	6	277 277 2009
TABLE	57E4 0165/hr	1-5-82	275	350	250	0 0	603
	465 W=100	1-5	174 174 880	1.11.1	1 1	0	2.0
•	AVCANGE STEU (Steam Flow = 10016s/hr,	11-4-12	698	1111	VV	8	707 707 707
		DATE	furnace Temp oc Te-1 Te-2	Bed Temp, oc TC-1 TC-3	70-5	TubE, Dec fres.	Shift Compasits Motot (22) VFA Density, 9/ml lodine No.
; •			1	4	· -	. H	· · · · · · · · · · · · · · · · · · ·

APPENDIX B



TABLE A-1 REGEN.

PORE VOLUME BY MERCURY INTRUSION AND NITROGEN DESORPTION

RADII RANGE ANGSTROMS	INTRUSION Ha INTRUSION	AN 82-3 N2 DESORFTION
		PORE VOLUME ML./G.
50,000 - 39,800	0.0301	and the state of t
39,800 - 25,100	0.0230	
25,100 - 15,800	0.0010	
15,800 - 10,000	0.0040	
10,000 - 6,300	0.0067	
6,300 - 3,980	0.0079	
3,980 - 2,510	0.0052	
2,510 - 1,580	0.0100	
1,580 - 1,000	0.0821	
1,000 - 630	0.0105	
630 - 398	0.0118	i
398 - 251	0.0064	0.0075
251 - 158	0.0243	0.0101
158 - 100	0.0150	0.0104
100 - 63	0.0722	0.0321
63 - 39.8	0.0556	0.0720
39.8 - 35	0.0151	
39.8 - 25.1		0.1187
25.1 - 15.8	· · · · · · · · · · · · · · · · · · ·	0.1753
15.8 - 10.0		0.0626

TABLE B-1

REGEN.

CUMULATIVE FORE VOLUME IN ALL FORES HAVING RADII LESS THAN THE INDICATED SIZE *

FORE RADIUS ANGSTROMS	LAB NUMBE 8 JAN 82-
50,000	0.8250
39,800	0,7949
25,100_	0.7719
15,800	0.7709
10,000	0.7669
6,300	0.7602
3,980	0.7523
2,510	0.7471
1,580	0.7371
1,000	0.6550
630	0.8445
398	0.6327
251	0.6263
158	0.6020
100	0.5870
63	0.5549
39.8	0.4829
25.1	0.3642
15.8	0.1889
10	. 0.1263
	▼

* DATA LESS THAN 100 ANGSTROMS FROM NITROGEN DESORPTION

TABLE C-1

PORE VOLUME DISTRIBUTION DATA *
LAR NO. 8 JAN 82-3 REGEN

:===========			\	
PORE RADII IN	ANGSTROMS	FORE VOLUME, ML./G. DRY BASIS	CALC. SURFACE DRY E	
		INCREMENTAL		CUMULATIV
50,000 - 39,800	39,800	0.0301	0.013	0.013
39,800 - 25,100	25,100	0.0230	0.014	0.028
25,100 - 15,800	15,800	0.0010	0.001	0.029
15,800 - 10,000	10,000	0.0040		0.035
10,000 - 6,300		0.0067	0.016	0.051
6,300 - 3,980	3,980	0.0079	0.031	0.082
3,980 - 2,510	2,510	0.0052	0.032	0.114
2,510 - 1,580	1,580	0.0100	0.098	0.212
1,580 - 1,000	1,000	0.0821	1.273	1.485
1,000 - 630	630		0.258	1.742
630 - 398	398	0.0118	0.459	
398 - 251	251	0.0064	0.394	2.596
251 158	158		2.377	
158 - 100	100		2.326	7.298
100 - 63	63		7.84	15.13
63 - 39.8	39.8		27.86	43.00
39.8 - 25.1	25.1	0.1187	72.80	
25.1 - 15.8	15.8		170.54	286.33
15.8 - 10.0	10.0		96.60	382.24
RESIDUA	L SURFACE AREA	•		256
				(
* DATA LESS THAN	100 ANGSTROMS	FROM NITROGEN DESORFT	ION	4
		- : := = = : : = = = :		/a

E-2-35

TABLE A-2

y

FORE VOLUME BY MERCURY INTRUSION AND NITROGEN DESORPTION

RADII RANGE ANGSTROMS	LAB NO. 21 JA Hs INTRUSION	N 82-1 N2 DESORFTION
	INCREMENTAL D.B. M	
50,000 - 39,800	0.0277	
39,800 - 25,100	0.0007	
25,100 - 15,800	0.0025	
15,800 - 10,000	0.0015	
10,000 - 6,300	0.0023	
6,300 - 3,980	0.0208	
3,980 - 2,510	0.0113	
2,510 - 1,580	0.0524	
1,580 1,000	0.0140	
1,000 - 630	0.1129	
630 - 398	0.0075	
398 - 251	0.0088	0.0086
251 - 158	0.0296	0.0142
158 ~ 100	0.0781	0.0168
100 - 63	0.0234	0.0386
63 - 39.8	0.0882	0.0773
39 . 8 - 3 5	0.0189	
39.8 - 25.1		0.1317
25.1 - 15.8		0.1775
15.8 - 10.0		0.0422

VIRAIN

CUMULATIVE FORE VOLUME IN ALL FORES HAVING RADII LESS THAN THE INDICATED SIZE *

	RADIUS TROMS	Lab Numbi 21 Jan 8:		
50,	000	0.9220	00 th no on to so so so, of the sh no so so so so	
39,	800	0.8943		
	100	0.8936		
	800	0.8911		_
10:	000	0.8896		
6:	300	0.8873		
	980	0.8665	····	_
2:	510	0.8552		
	580	0.8028		
1	000	0.7888		_
	630	0.6759		
	398	0.6684		
•• •	251	0.6596		
	158	0.6300		
	100	0.5519		
	63	0.5133		
-	39.8	0.4360		
	25.1	0.3043		
- Pr cape	15.8	0.1268		
•	10	0.0846		
	• •	, 0,000		

^{*} DATA LESS THAN 100 ANGSTROMS FROM NITROGEN DESORPTION

TABLE C-2

VIRGIN

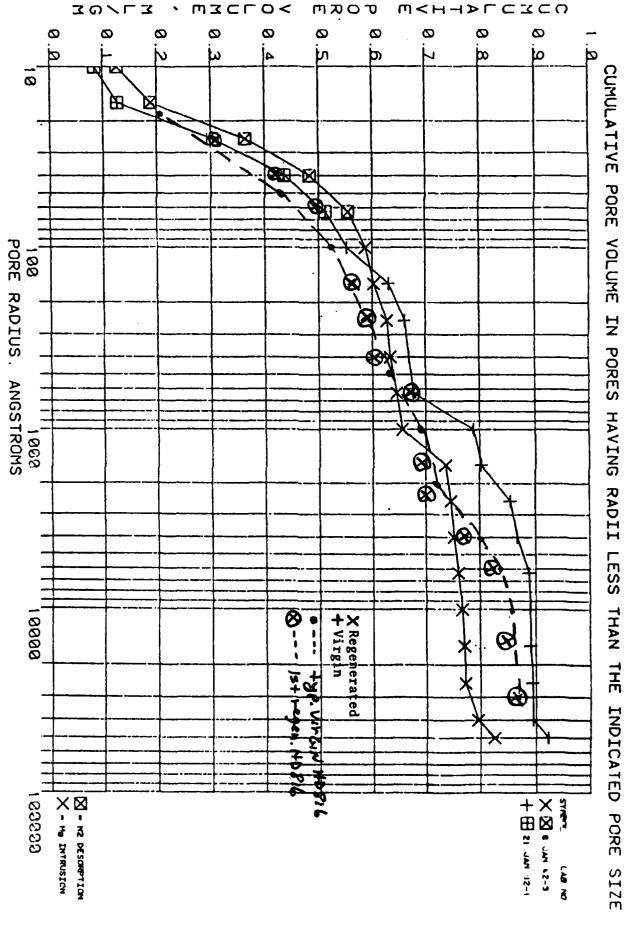
FORE VOLUME DISTRIBUTION DATA *

FORE RADII IN	ANGSTROMS	PORE VOLUME, ML./G. DRY BASIS	CALC. SURFACE DRY BO	
INCREMENTAL	CUMULATIVE	INCREMENTAL	INCREMENTAL	CUMULATI
50,000 - 39,800	39,800	0.0277	0.012	0.012
39,800 - 25,100	25,100	0.0007	0.000	0.013
25,100 - 15,800	15,800	0.0025	0.002	0.015
15,800 - 10,000	10,000	0.0015	0.002	0.018
10,000 - 6,300	6,300	0.0023	0.006	0.023
6,300 - 3,980	3,980	0.0208	0.081	0.104
3,980 - 2,510	2,510	0.0113	0.070	0.174
2,510 - 1,580	1,580	0.0524	0.512	0.686
1,580 - 1,000	1,000	0.0140	0.217	0.903
1,000 - 630	630	0.1129	2.771	3.674
630 - 398	378	0.0075	0.292	3.966
398 - 251	251	0.0088	0.542	4.508
251 - 158	158	0.0296	2.895	7.403
158 - 100	100	0.0781	12.109	19.511
100 - 63	63	0.0386	9.43	28.94
63 - 39.8	39.8	0.0773	29.91	58.8 <i>£</i>
39.8 - 25.1	25.1	0.1317	80.72	139.58
25.1 - 15.8	15.8	0.1775	172.66	312.24
5.8 - 10.0	10.0	0.0422	65.12	377.37
RESIDUA	L SURFACE AREA	A		213

* DATA LESS THAN 100 ANGSTROMS FROM NITROGEN DESORPTION



The second of th



3531 353555 353576

The second secon

A. P. Forrard SC Brud Do.

TABLE A-1

RADIT RANGE	LAT NO. 8 JA	
ands trums	INTRUSTUR	METT 4000 20
		PORE VOLUME
:0+000 - 39+800 .	0.0248	s that about the to be that and the the course of the course
19,000 - 25,100	0.0137	
5,:00 - 15,200	0.0035	
5,800 - 10,000	0.0174	
0.000 " 3.300	0.0122	
61300 - 3198 6	0.0109	
Jr980 - 2,510	0.0200	
J. 010 - 1,580	0.0172	
1,580 - 1,000	0.0134	
17000 - 630	0.0790	
იკი - კემ	0.0341	
398 - 251	\$\\\$405	U.0038
251 - 158		0.0167
158 - 100	0.0138	0.0220
10 0 - 43	0.0023	0.0304
63 · 39.2	0.0707	0.0509
37.8 + 35	0.0077	•
77.4 - 25.1		0.00333
ាធិលា 😁 🚉 💆 💎	· ·	0.1390
15.8 - 10.0	SPAP NAME I	以,锁锁设施

TABLE B-1

CUMULATIVE PORE VOLUME IN ALL PORES HAVING RADII LESS THAN THE INDICATED SITE*

Pore Radius	Incremental Pore Volume
Angstroms	D.B. ml/g
50,000	0.7580
37,800	0.7332
25,100	0.7195
15,800	0.7160
10,000	0.6986
6,300	0.6864
3,980	0.6708
2,510	0.6555
1,580	0.6303
1,000	0.6199
630	0.5909
390	0.5568
251	0.5163
150	0.4985
100	0.4847
63	0.4548
39.0	0.3974
25.1	0.3139
15.8	0.1741
10	0.0437

^{*} Data less than 100 angstroms nitrogen desorption.

TABLE B-1

FUMULATIVE PART VOLUME IN ALL PARTS HAVING RADII (FAS THAN THE INDICATED SITE (

ं वा प्रशासन क्षात्र का का का का का का प्रशासन कर है	(加速型表现有基础整理人),及型类型的人。以及其特殊人员的
HOSE RADIUS	LAR NUMBER
ANDS INDES	9 JAN 82 4
The second secon	0.7590
50,000	
39+800	0.7332
2011/00	0.7495
15,800	0.7160
10.000	0.6986
son	0.6844
1.980	0.67%3
1.410	0.4555
11580	0.6793
1.000	9.6190
430	0.5000
***;	0.5568
<u> </u>	0.5 333
<u> </u>	0. 4986
100	0.4847
63	0.4%43
<i>39</i> (1	0.3974
25.3	0.3134
	0.1741
10	0.043
, U	** * * * * * * * * * * * * * * * * * *

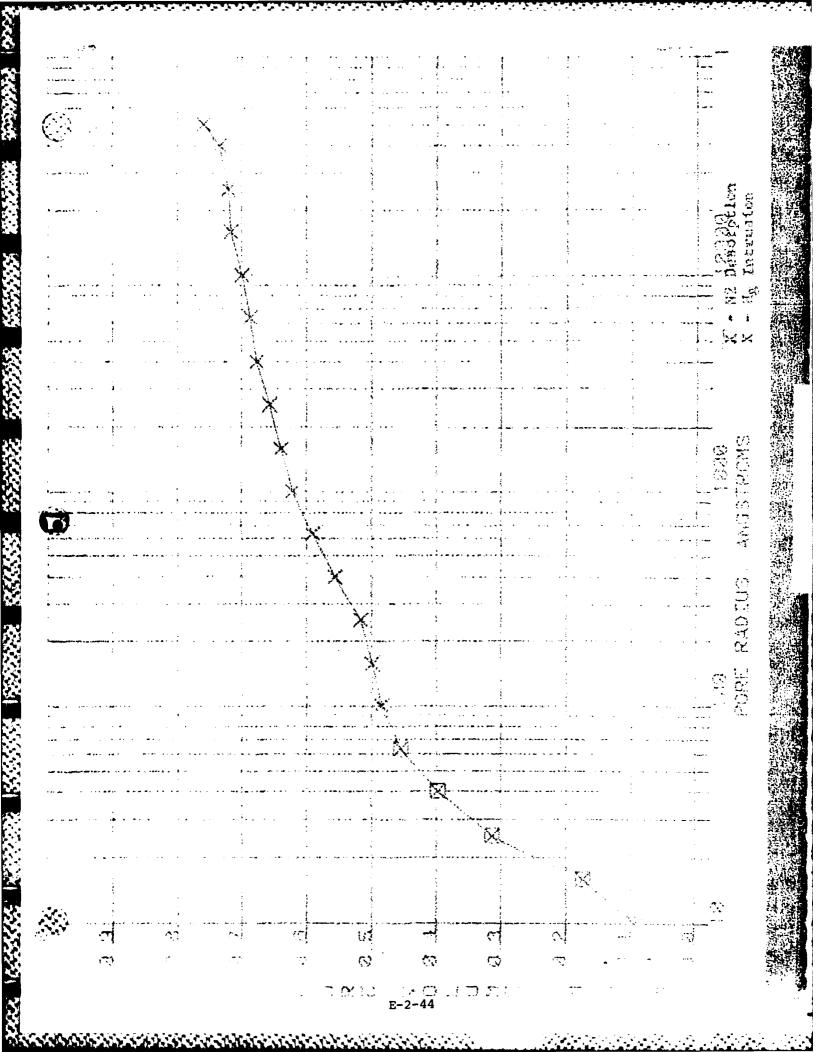
^{*} DATA CERS HAN 100 ANDREWERS - DES MITROPORT TOURFTION

TAME C-1

FORE VOLUME BIBIRIESTION DATA & LAB NO. & JAN 62-4

PORE BADIS IN	AMBRIRUMS	PURE VOLUME, ML./6. BKY BASIS	CALCA SURFACE 587 E	
THUREMENTAL	CUMULATIVE	INCEPENTAL	MERCHENTAL	in in a second
50:000 - 79:000	39,000	0.0248	6 41	
37.800 Jt. 100	25.100	449137	V.008	· .
	12,800	4.003%	⊕,0 03	; :
15.800 - 10.000	10.000	0.0174	0 x 0 2 7	ر.
(d),000 - 2+100	4,300	0,0122	0 0 3 0	C'e
6.300 - 3.400	780ءڌ	0.0109	0.442	٠.
	2,510	6.0200	0.123	,
2.510 . 1.530	1:580	2.0172	U • 1.66	14
1.500 - 1.000	1,000	0.0184	0 , 285	<u>;</u>
1,000 000	450	0.0596	0.71 2	:
530 · 390	398	0.0341	1 - 327	•
399 - 251		0.0405		5
251 150	158	0.0178	1.741	69
158 - 100	100	1 1. 1.0138	2-140	9
100 - 65	63	U.0304	7.42	16
63 × 39.18	39 .8	৽৽৽৽৽৽৽৽৽৽৽৽৽৽৽৽৽৽৽৽৽৽৽৽	72.01	38
30.8 - 54.1	25.1	ಿ , ೧೦೮೩	51.20	89
Training the state of	19.0	0,1398	13598	225
10,8 - 10,0	10,0	0.0804	12400	349
1668 Fre194	. SHEFACE AREA			119

^{*} DATA LEGG THAN TOO ANGSTROMS FROM MITROGEN DECORPTION



SECTION 3

INORGANIC ANALYSES FOR SETTLED SLUDGE SAMPLES

TABLE E-3-1 SETTLED SLUDGE MAJOR CATIONS, ANIONS, AND NUTRIENTS

	Phase IA & IB Settled Sludme	Phase IIA Settled Sludge	
Calcium			
(MDL= 0.2 ms/1)	301	52	
No. of Samples No. Above MDL	301	52 52	
Arithmetic Mean	89.06	15734.90	
Standard Deviation	72.32	16687.48	
Geometric Mean Spread Factor	71.13 1.89	8319.76 4.00	
Median Value 90% Less Than	65.0 176.0	9200.0 39000.0	
Hardness: by addition (Ca+Ms. as CaC)3)		
(MDL= 1.0 mg/1 CaCD3)			
No. of Samples No. Above MDL	301 301	52 52	
Arithmetic Mean	325.8	53103.0	
Standard Deviation	248.5	599 32.2	
Geometric Mean Spread Factor	270.0 1.77	25953.8 4.25	
Median Value 90% Less Than	244 577	26673 129569	
Magnesium			
(MDL= 0.1 ms/1)		==	
No. of Samples No. Above MDL	301 301	53 53	
Arithmetic Mean Standard Deviation	25.12 20.86	2183.65 2480.85	
Geometric Mean Spread Factor	21.07 1.71	1083.88 3.49	
Median Value 90% Less Than	19.8 36.0	950.0 6300.0	
Potassium			
(MDL= 0.3 ms/1)			
No. of Samples No. Above MDL	300 300	53 53	
Arithmetic Mean Standard Deviation	12.94 14.62	48.08 67.92	
Geometric Mean Spread Factor	10.27 1.85	17.29 4.30	
Median Value 90% Less Than	9.8 16.3	9.0 190.0	
Sodium			
(MDL= 0.1 ms/1)			
No. of Samples No. Above MDL	301 301	53 53	
Arithmetic Mean Standard Deviation	31.47 21.19	274.89 467.40	
Geometric Mean Spread Factor	27.88 1.57	72.76 4.78	
Median Value	28.0	42.0	
90% Less Than	41.5	1100.0	

TABLE E-3-2 SETTLED SLUDGE TRACE METALS

THE THINKS SOUTH THE THE THE STATE OF THE ST

	Phase IA & IB Settled Sludse	Phase IIA Settled Sludse	
 Aluminum (MDL= 0.003 ms/1)			
No. of Samples, No. Above MDL	283 282	53 44	
Arithmetic Mean Standard Deviation	1219.0920 1290.9122	62.4154 171.7769	
Geometric Mean Spread Factor	741.5954 3.42	2.5454 86.33	
Median Value 90% Less Than	683.000 2800.000	25.000 78.000	
 Antimony			
(MDL= 0.0003 ms/1)			
No. of Samples No. Above MDL	269 124		
A-111 M			
Arithmetic Mean Standard Deviation	0.03578 0.09025		
Geometric Mean Spread Factor	0.00034 135.88		
Median Value	ND		
90% Less Than	0.0800		
 Arsenic	*********************	***********************	
(MDL= 0.0002 mg/1)			
No. of Samples No. Above MDL	300 243	53 43	
Arithmetic Mean Standard Deviation	0.2 99 02 0.273 9 0	0.02766 0.04908	
Geometric Mean	0.05250	0.00592	
Spread Factor	36.00	11.18	
Median Value 90% Less Than	0.2600 0.6600	0.0100 0.06 5 0	
Barium (HDL= 0.002 mm/1)			
No. of Samples No. Above MDL	297 293	5 2 5 1	
NO. HOUVE TIDE	273	31	
Arithmetic Mean Standard Deviation	1.2758 1.1405	2.3746 1.8667	
Geometric Mean Spread Factor	0.8893 3.02	1.7334 3.16	
Median Value 90% Less Than	1.030 2.300	1.800 4.200	
 Beryllium			
(MDL= 0.0008 ms/1) No. of Samples	247		
No. Above MDL	267 0	•	
Arithmetic Mean	ND		
Median Value	ND		

	Phase IA & IB Settled Sludme	Phase IIA Settled Sludge	
Boron			
(MDL= 0.0040 ms/1)	•		•
No. of Samples	299	53	
No. Above MDL	70	50	
Arithmetic Mean	0.07846	0.62108	
Standard Deviation	0.21 899	0.52673	
Geometric Mean	0.0009	0.37304	
Spread Factor	276.30	4.16	
M- 41 44 4		A Mana	
Median Value 90% Less Than	ND 0.2200	0.5000 1.1200	
YOU LESS THEIR	0.2200	111200	
	,		
Cadmium: ICAP (MDL= 0.0008 mg/1)			
No. of Samples	247		
No. Above MDL	235		
Anishmatic Moon			
Arithmetic Mean Standard Deviation	0.16056 0.12087		
Geometric Mean	0.10645		
Spread Factor	3.77		
Median Value	0,1380		
90% Less Than	0.2900		
Cadmium: furnace AAS			
(MDL= 0.0002 ms/1)			
No. of Samples	53	53	
No. Above MDL	23	6	
Arithmetic Hean	0.00860	0.00116	
Standard Deviation	0.01408	0.0 039 7	
Geometric Mean	0.00016	Not	
Spread Factor	87.50	Calculated	
	3,,33	33.33.33	•
Median Value	ND	ND	
90% Less Than	0.0250	0.0003	
Chromium: ICAP (MDL= 0.003 mg/1)			
No. of Samples	247		
No. Above MDL	238		
And About 1 - Many	A 5041		
Arithmetic Mean Standard Deviation	0.5091 0.6606		
Geometric Mean	0.3235		
Spread Factor	3.30		
Median Value	0 .38 0		
90% Less Than	0.820		
Chromium: furnace AAS			
(MDL= 0,0002 ms/1)			
No. of Samples No. Above MDL	51 50	52 42	
NO. HOOVE FILE		74	
Arithmetic Mean	0.44204	0.27 83 2	
Standard Deviation	0.62772	0.35891	
Geometric Mean	0.21360	0.02716	
Spread Factor	4.65	. 38.80	
		-	
Median Value 90% Less Than	0.2600 0.9 5 00	0.1500 0.6900	

		'Phase IA & IB Settled Sludse	Phase IIA Settled Sludse
	Cobalt: ICAP (MDL= 0.003 ms/!) No. of Samples No. Above MDL	248 116	
	Arithmetic Mean Standard Deviation	0.0602 0.07 89	
	Geometric Hean Spread Factor	0.0037 34.16	
	Median Value 90% Less Than	ND 0.160	·
	Cobalt: furnace AAS (MDL= 0.0001 mg/1)		***************************************
	No. of Samples No. Above MDL	21 21	
	Arithmetic Mean Standard Deviation	0.35015 0.79625	
	Geometric Mean Spread Factor	0.15141 3.48	
	Median Value 90% Less Than	0.2000 0.3100	
W	Coppert ICAP (MDL= 0.0008 ms/1) No. of Samples	248	
	No. Above MDL Arithmetic Mean	239 0.48903	
	Standard Deviation	0.30158	
	Geometric Mean Spread Factor	0.33960 3.93	
	Median Value 90% Less Than	0.4130 0.9000	
*****	Copper: flame AAS (MDL= 0.0012 mg/1) No. of Samples No. Above MDL	53 53	53 48
	Arithmetic Mean Standard Deviation	0.56155 0.29262	0.40074 0.43848
	Geometric Mean Spread Factor	0.48561 1.79	0.16743 7.55
	Median Value 90% Less Than	0.5200 1.0000	0.3200 0.7400
	Iron (MDL= 0.003 ms/1)		
	No. of Samples No. Above MDL	292 2 9 2	46 41
	Arithmetic Mean Standard Deviation	123.3864 92.9567	58.4476 112.8975
(iii)	Geometric Mean Spread Factor	89.8721 2.54	6.1236 46.21
	Median Value 90% Less Than	105.000 230.000	28.000 144.000

والمراكبين والمجارون والمجارون المجاولات المجارون المجارون والمراجعة

	TABLE E-3-2 SETTLED SLUDGE TRACE METALS (Continued)	
	Phase IA & IB Settled Sludse	Phase IIA Settled Sludge
Lead		
(MDL= 0.0003 ms/1) No. of Samples No. Above MDL	295 280	53 37
Arithmetic Mean Standard Deviation	0.15730 0.19233	0.28498 0.55936
Geometric Mean Spread Factor	0.07 47 3 5.78	0.01178 75.01
Median Value 90% Less Than	0.1050 0.3500	0.0720 0.7000
Lithium: ICAP		,u,a,_,~?===,u==,u=
(MDL= 0.0010 ms/l) 'No. of Samples No. Above MDL	245 219	
Arithmetic Mean Standard Deviation	0.07696 0.06758	
Geometric Mean Spread Factor	0.04113 5.06	
Median Value 90% Less Than	0. <i>0</i> 630 0.1500	
Lithium: flame AAS		
(MDL= 0.0004 ms/1) No. of Samples No. Above MDL	53 50	53 41
Arithmetic Mean Standard Deviation	0.14092 0.27533	0.04728 0.08022
Geometric Mean Spread Factor	0.05873 5.12	0.00 98 7 14.08
Median Value 90% Less Than	0.0650 0.3300	0.0300 0.0950
Manganese		
(MDL= 0.0010 ms/1) No. of Samples No. Above MDL	286 286	53 52
Arithmetic Mean Standard Deviation	152.59343 212.05031	15.59661 12.36074
Geometric Mean Spread Factor	51.99530 5.26	7.96604 6.27
Median Value 90% Less Than	64.3000 419.0000	14.5000 34.0000
Mercury		
(MDL= 0.00027 ms/1) No. of Samples No. Above MDL	299 283	53 42
Arithmetic Mean Standard Deviation	0.00213 0.001 5 0	0.00128 0.00105
Geometric Mean Spread Factor	0.00169 2.11	0.00086 2.77
Median Value 90% Less Than	0.0018 0.0039	0.0011 0.0026





		Phase IA & IB Settled Sludge	Phase IIA Settled Sludwe	
	Molybdenum			
	(MDL= 0.002 mm/1) No. of Samples No. Above MDL	266 7		
	Arithmetic Mean Standard Deviation	0.0067 0.049 5		
	Median Value 90% Less Than	ND ND		
	Nicke? (MDL= 0.0010 mg/l)		*****************************	
	No. of Samples No. Above HDL	29 4 291	53 46	
	Arithmetic Mean Standard Deviation	0.33798 0.64929	0.30478 0.25552	
	Geometric Mean Spread Factor	0.20046 2.83	0.11383 10.06	
	Median Value 90% Less Than	0.2200 0.5730	0.2500 0.6500	
	Selenium (MDL= 0.0002 mg/l)			
. .	No. of Samples No. Above MDL	301 75	53 11	
9	Arithmetic Mean Standard Deviation	0.00915 0.05157	0.00997 0.04854	
	Geometric Mean Spread Factor	0.00000 527.87	0.00000 656.95	
	Median Value 90% Less Than	ND 0.0223	NB 0.0150	
	Silver: flame AAS (MDL= 0.0008 mg/l)	#**		
	No. of Samples No. Above MDL	248 27		
	Arithmetic Mean Standard Deviation	0.00368 0.01215		
	Median Value 90% Less Than	ND 0.0200		
	Silver: furnace AAS (MDL= 0.0002 mg/1)			
	No. OF Samples No. Above MDL	53 36	53 33	
	Arithmetic Mean Standard Deviation	0.01426 0.02142	0.00700 0.01144	
	Geometri i Mean Gentini Sulan	0.00181 19.71	0.00088 18.68	
	Modern Calle 20% to see Tra	0.00 50 0.0 45 0	0.0050 0.0150	

LASSASS LOCADOR DESIGNAS RECESSOS RECESSOS

	Phase IA & IB Settled Sludse	Phase IIA Settled Sludse	
Thallium (HDL= 0.0009 me/l) No. of Samples No. Above HDL	269 2		~~~~
Arithmetic Mean Standard Deviation	0.00308 0.04146		
Median Value 90% Less Than	ND ND		
Tin (MDL= 0.0040 mm/l) No. of Samples No. Above MDL	264 110		
Arithmetic Mean Standard Deviation	0.16904 0.33476		
Geometric Mean Spread Factor	0.0024 9 86.97		
Median Value 90% Less Than	ND 0.5300		
Titanium (MDL= 0.0020 ms/1) No. of Samples No. Above MDL	297 280	53 16	
Arithmetic Mean Standard Deviation	0.812 8 2 0.75513	0.29372 0.82095	
Geometric Mean Spread Factor	0.42 95 7 5.36	0.00009 954.72	
Median Value 90% Less Than	0.6400 1.6700	ND 0.8000	
Vanadium (MDL= 0.0020 mg/l) No. of Samples No. Above MDL	2 98 273	53 36	
Arithmetic Mean Standard Deviation	0.26674 0.2120 5	0.16287 0.21992	
Geometric Mean Spread Factor	0.14780 5.10	0.02420 21.86	
Median Value 90% Less Than	0.2300 0.5100	0.1000 0.3600	
Zinc: ICAP (MDL= 0.0020 mg/1) No. of Samples No. Above MDL	248 247		
Arithmetic Mean Standard Deviation	2.24500 1.53561		
Geometric Mean Spread Factor	1.86051 2.02		
Median Value 90% Less Than	1.9000 3.9400		



	Phase IA & IB Settled Sludse	Phase IIA Settled Sludse	
Zinc: flame AAS			
(MDL= 0.0012 mg/1)			
No. of Samples	53	53	
No. Above MDL	53	41	
Arithmetic Mean	3.31774	1.41263	
Standard Deviation	1.72636	1.55931	
Geometric Mean	2.81827	0.16041	
Spread Factor	1.92	46.69	
Median Value	3.1000	1.1000	
90% Less Than	5.5500	3.2000	
•			

SECTION 4

PERFORMANCE RESULTS FOR THE REMOVAL OF VOLATILE ORGANIC CHEMICALS BY SURFACE AERATION

Table E.4-1 presents results from a special testing program conducted to assess the removal of several volatile organic chemicals from the EEWTP blended influent by mechanical surface aeration. The test was conducted on 24 and 25 June 1982 and consisted of analyses (by LLE) of influent and effluent aeration tank samples taken hourly over a twenty-four hour period. A detailed description of the 2 hp mixer and aeration tank are provided in Chapter 7, Section 1.

The results indicate that of the six compounds detected in the blended influent only two, chloroform and tetrachloroethene, were removed to a significant level by mechanical aeration process.

Performance Results for the Removal of Volatile Organic Chemicals by Surface Aeration

SURFACE AERATION PERFORMANCE VOLATILE ORGANIC CHEMICALS (BY LLE PHASE 1¹

COLOROR PRODUCT STANDARD STANDARD STANDARD

AND TO STATE WASHING BELLING TOWNS STATES

•	Aer	Aerator Influent			Aerato	Aerator Effluent		Percent Removal	95% Confidence Interval	idence
Z	No.2 Detected	Geometric	Spread	z	No. Detected	Geometric	Spread Factor	×	Lower	Upper
Chlorofrom 24 Bromodichloromethane 24 Dibromochloromethane 24 Bromoform 24 Total THMs 24 Trichloroethane (TCE) 24 Trichloroethane (TCE) 24 Trichloroethane (TCE) 24	778°72	1.55 0.18 NC3.097 1.85 0.10	1.23 1.85 1.86 NC 1.32	*********	772"77	1.17 0.15 0.095 NC 1.43 0.22	1.42 1.92 1.89 NC 1.48	25 2 2 - 23 -120	10.8 -21.7 -51.9 - 5.9 -209	36.1 42.9 36.8 -56.6
	\$	96.0	1.27	2	3	25.0	1.31	42	33.0	20.5

Results from 24 grab samples collected at influent and effluent locations hourly between 0800 on 24 June and 0700 on 25 June 1982.

Water temperature = 24°C.

MDL of 0.1 mg/L assumed for all analyses (conducted on-site).

Insufficient quantified samples: Not Calculated.

FILMED

2 = 34